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THE INSTITUTE OF PETROLEUM

An Ordinary General Meeting of the Institute of Petroleum was held at Manson House, 26 Portland Place, London, W.1, on October 10, 1951, Mr J. S. Parker, vice-president, presiding.

The Chairman conveyed apologies from the president, Mr C. A. P. Southwell, who was in Kuwait, for being unable to attend, and his best wishes for a successful evening.

The Minutes of the Ordinary General Meeting, held on September 19, 1951, were read, confirmed, and signed.

A list of new members elected to the Institute was tabled.

The Chairman, introducing the authors of the papers to be presented, said: A major problem to oil refiners, more especially those interested in the acid refining of lubricating oils, has been the efficient disposal of the resultant waste product, known to them as acid sludge.

This problem is general to all oil refiners, and the acid sludges produced are, in general, of similar qualities. Each oil refiner has had a similar problem which he has developed according to his own ideas. It is thus of interest to note how each refiner has attacked the problem, and this symposium has been organized, in so far as the papers to be discussed are concerned, for that purpose.

Each oil refiner has his own little secrets, but on general problems, such as acid sludge, there has always been co-operation and a free interchange of information. The papers which are being presented for discussion are fully representative of the present lubricating-oil refining capacity of the U.K.

These papers are factual and cover the present methods used by the respective companies. The methods are, in the main, concerned with recovering the fuel value of the acid sludges. Lately a further problem has been added, that of sulphur conservation. Discussion will be of the greatest advantage in dealing with this point.

The following papers were then presented in summary.

ACID SLUDGE—ITS UTILIZATION AND DISPOSAL

By A. Cluer * (Member)

Introduction

This contribution is confined to the consideration of the acid sludge problem as faced by one of the smaller British refineries, producing a complete range of petroleum products and having a total annual throughput of approximately 150,000 tons.

* Lobitos Oilfields Ltd.

Sulphuric acid is not used in the refining of any of the atmospheric products; of the lubricating oils manufactured, a considerable proportion is refined solely by solvent treatment (Edeleanu process). Hence, acid sludge results only from a proportion of the lubricating oil output, about 18,000 tons per annum being acid treated.

Such treatment is carried out in three plants :—

A. Batch agitators (capacity *ca* 40 tons oil), used for refining various oils in a wide viscosity range (about 4 to 400 cs at 60° C); by far the greater proportion of the oil treated is, however, of the heavier grades.

B. Continuous treatment of technical white oils and medicinal oils in a plant employing centrifugal separation of sludge and oil.

C. Continuous treatment (at higher temperatures, up to about 100° C) of industrial oils (viscosity up to 400 cs at 60° C) in a plant employing centrifugal separation. Treatment in this plant is eventually intended to replace much of the processing at present handled in plant A above.

Plants B and C were commissioned in 1949 and 1951 respectively, hence the acid sludges produced prior to this period (mainly derived from treatment with acid of 95 per cent strength) were mostly of the heavy lubricating oil type, which weather to a solid material on exposure to the air in open pits.

Laboratory trials showed that it was technically possible to mix this sludge with fuel oil and burn it, but the economics were unfavourable as compared with dumping. Burning of the sludges in the solid form was not feasible in the absence of any coal-fired boilers in which this could be accomplished. Other schemes for the disposal of solid sludges which were given laboratory or pilot-plant trials included pulverizing, followed by water washing and briquetting, and also coking (at about 180° C) to drive off acid.

This latter procedure resulted in a brittle lustrous hard material with a m.p. of 95° C and containing only 4 to 5 per cent of matter insoluble in carbon disulphide. Possible uses of this, either as a fuel or for blending with bitumen, were investigated, but, like the other procedures suggested for utilizing these solid sludges, both technical and economical difficulties were encountered.

Prior to the commissioning of the new plants mentioned above, the problem of dealing with the mainly solid sludges was never sufficiently acute to warrant pursuing either the question of burning or disposing in a manner other than by dumping. The latter procedure was therefore continued for many years, the sludge being dug out of the storage pits and transported by road to local tipping sites.

Present-day Position

With the bringing into operation of the continuous treatment plants, involving centrifugal separation of sludge and oil, the situation changed. The quantities of oil to be treated, and hence the quantities of sludge produced, were much increased, and sludge resulting from the newer processes was relatively easily pumpable.

The quantities and typical qualities of all the various sludges available are shown in Table I.

Plant	Type of oil treated	Acid intake, tons/year	Sludge produced, tons/year	Estimated sulphur content of sludge, tons/year	Typical properties of sludge		
					Sp. gr.	Water, %	Titratable H_2SO_4, % wt
B	Technical white	1200 (20% oleum)	1600	350	1·5	3	60–70
B	Liq. paraffin	2800 (20% oleum)	3400	750	1·3–1·7	2·5	50–80
A, C	Industrial	500 (95% acid)	500 *		1·8	3	92
		500 * (liq. par. sludge)	2500	300	1·2–1·3	5–6	25–40

* Transferred to and included in A, C.

Approximately 1500 tons of sulphur is contained in the fresh acid feeds indicated in Table I, and it will be seen that 1400 tons of sulphur is estimated to be present in the sludges. The remainder of the sulphur appears as sulphonic acids in the acid oils, or is lost to atmosphere as sulphur dioxide during the course of the reaction.

It will be noted that the high (90 per cent) acidity mobile sludge which is obtained (at a rate of about 500 tons/year) in the final stages of the manufacture of medicinal oil, is taken as acid feed for the batch treatment of industrial oils, being thereby degraded to the solid type of sludge, and thus forming part of the total of 2500 tons/year of sludge which is dumped.

This figure includes the sludge yielded by the continuous acid treatment plant for the processing of industrial oils (plant C). This sludge, being produced at a higher temperature than when similar oils are batch treated in agitators, is considerably more fluid than in the latter case, and should be burnable either by itself or else after mixing with either fuel oil or the more mobile oleum sludge produced on plant B.

However, the industrial oil continuous treatment plant, having only just been put into operation, has not yet settled down to a regular routine schedule and, for the time being, the sludge coming from it is being disposed of, along with that from the agitators, by dumping. The balance of 5000 tons/year of sludge produced from the white oil plant B is at present disposed of by burning, although, as will be mentioned later, the possibility of obtaining useful materials from the sludge prior to such burning has been investigated and has shown promising results.

Sludge Disposal by Burning

Two systems for burning mobile sludges were installed. The capacity of each system alone is sufficient to cope with the sludge output of the white oil plant, the reserve capacity being made available in case of breakdown of one system, and for future handling of fluid sludges from plant C.

The first system involves pumping the sludge 700 ft to a receiving tank and thence to a Dutch oven located alongside an existing battery of Economic boilers. Two sludge burners and one auxiliary oil burner are installed in this oven, and the resulting hot flue gases, discharged to one of the boilers, raise steam at 80 p.s.i.g. Early sludge burners employed steam atomization, but corrosion of the burners was very rapid. A

simple burner using air atomization, with a $\frac{3}{8}$-inch dia tip of stainless steel (18% Cr, 8% Ni, 3% Mo) was found satisfactory, the tip lasting several months. The dimensions of the oven are 14 ft long by 7 ft wide by 8 ft high, and in four months of fairly continuous operation it disposed of about 1000 tons of mobile sludges, raising 2600 tons of steam. Maximum disposal rate is approximately 15 tons/day, giving an estimated heat release of about 9000 B.Th.U/hr/cu. ft. of furnace volume, assuming a calorific value of 5000 B.Th.U/lb for the sludge.

Published figures concerning optimum heat release for a given furnace volume when burning acid sludges vary widely from 4000 [1] to more than

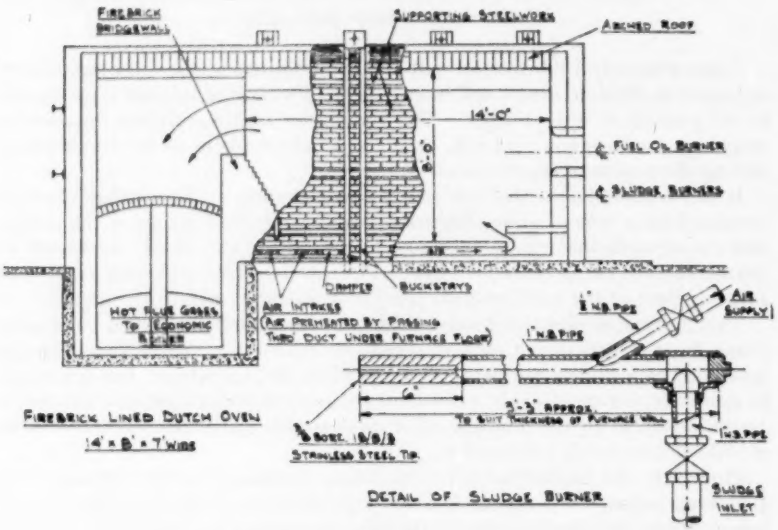


FIG. 1

40,000 [2] B.Th.U/hr/cu. ft. of furnace volume, depending on the calorific value of the sludge being handled.

The brickwork of the Dutch oven suffered appreciably from the combined effects of overheating and contact with acid gases. Part of this trouble was attributed to the quality of the firebrick available when the oven was constructed, this brick having an alumina content of only 28 per cent. The most severely affected parts of the lining have been renewed with 38 per cent alumina firebrick, but it is not as yet possible to estimate the effects of this change.

Fig. 1 gives a general indication of the Dutch oven and the sludge burner.

The second disposal system involves pumping the sludge 1400 ft to a receiving tank and thence to a pair of existing water-tube boilers, raising steam at 175 p.s.i.g. Each of these boilers was originally fired with five oil burners; a sludge burner was substituted for one oil burner in each combustion chamber. The flue gases from the two boilers discharge to a common steel stack 6 ft dia by 80 ft high, which was increased to a height of

100 ft and protected by a gunite lining prior to commencing sludge burning. The economizers were protected from the effects of acid flue gases by installing a pump to circulate water from the hot end of the economizer back to the cold end, thus maintaining feed water temperature to the economizers at around 180° to 200° F. In fact, due to insufficient preheating of the feed water, this temperature for some time averaged only 140° to 160° F, and slight corrosion of the economizer tubes took place, the coating of corrosion product on the surface of the tubes causing the economizer scrapers to stop working. Apart from this initial difficulty, no trouble in the boiler setting has been experienced in about a year's operation. The gunite stack lining has been inspected and found to be intact.

The sludge burners used in the water-tube boilers are of the same pattern as those used in the Dutch oven. Difficulties with atomization may occur with the more viscous sludges, particularly in cold weather, when there is a tendency for coke to build up in the combustion chamber. Any attempt to heat such sludges to reduce viscosity must be conducted with caution, in case of coking in the sludge tank and lines. Circulation of warm fuel oil through closed coils in the sludge receiving tank has been practised in this connexion with success.

The dilution of acidity in the stack gases by operating one sludge burner to four oil burners means that the stack gases from the water-tube boilers are rarely a nuisance. The stack gases from the Dutch oven can, however, at times be objectionable, particularly if a very fluid sludge of high H_2SO_4 content is being burned, and atmospheric conditions are such as to cause only slow dispersal of stack gases.

Mobile sludges are now normally disposed of by burning under the water-tube boilers, the Dutch oven equipment being held as standby and for future operation on heavier sludges of lower acidity and higher calorific value. Tests on acidity of stack gases from the water-tube boilers over a trial period of several days, burning sludges of about 50 per cent H_2SO_4 content at the rate they were being produced from the white oil plant, showed an average of 0·15 per cent volume SO_2/SO_3, equivalent to 2·3 grains SO_3/cu. ft. This agreed well with the calculated acidity based on the known quantities of sludge and fuel oil burned, and the excess air, which was of the order of 100 per cent, as measured by Orsat gas analysis.

Because it has been found necessary so far to use this comparatively large excess of air to burn the sludge completely, the overall efficiency of the water-tube boilers must to some extent be impaired. Nevertheless, when the first ten months' operation with sludge burning was compared with the previous 10 months prior to sludge burning, a net increase in steam raised per ton of fuel oil of about 7 per cent was proved. This is equivalent to about 2·8 tons of steam raised per ton of sludge burned, a figure very close to that quoted above for the Dutch oven.

Utilization of the Sludges Prior to Burning

That the acid sludges contain products of potential value is undoubted; whether, however, these can be economically recovered is another matter. In the case of the solid sludges, as has been mentioned, this has not been attempted, largely on account of handling difficulties and also because such

sludges contain less material of value than do the more mobile residues. In the case of the latter, however, pre-treatment before they are burnt has the dual advantage of recovery of usable products and also of reducing the acidity of the sludge, thus minimizing the nuisance from the flue gases resulting from its combustion.

Our investigations on the utilization of the mobile (oleum) sludges prior to their disposal by burning have been directed towards the recovery of two products, sulphur dioxide and sulphonic acids.

Sulphur Dioxide

The sludges discharged from the centrifuges in the white oil plant contain considerable quantities of SO_2, formed in the reaction between the oil being treated and the oleum used as a refining agent. This sludge is collected in receiving tanks in which, at a temperature of 40° to 50° C, a pressure of 3 to 4 p.s.i. above atmospheric, due to SO_2 can be built up. The gas above the sludge is free of SO_3, since the sludges always contain some H_2SO_4 of less than 100 per cent strength, in which any SO_3 is absorbed; moisture is also absent, since the sulphuric acid present is of sufficient concentration to retain any water.

Whilst it is necessary for comfortable operation of the white oil plant to ensure that the centrifuges operate at slightly below atmospheric pressure this can be combined with a pressure in the sludge-receiving tanks slightly above atmospheric. This is accomplished by always maintaining the inlet line into these tanks below the sludge level, thus forming a seal. Keeping the sludge tanks at a pressure slightly above atmospheric ensures that the very minimum amount of air is drawn into the Edeleanu plant, otherwise undue pressures would be experienced in the SO_2 condensers. A typical analysis of gas withdrawn from sludge tanks operating under these conditions is as follows :—

	% by wt
SO_2	98·7
SO_3	Nil
Water	0·3
Air (by difference)	1·0

Such gas is of sufficient purity to be drawn directly into the Edeleanu plant sulphur dioxide recovery system, making use of the vacuum pumps forming part of the Edeleanu plant equipment. The amount of dry SO_2 gas being normally handled by these pumps is sufficient to dilute the small amount of water, shown in the above analysis, to harmless proportions; the water is subsequently removed in the drying system forming part of the Edeleanu plant circuit.

The maximum SO_2 yield from the average oleum sludge appears to be about 3 to 4 per cent by weight. This figure has been determined by the loss in weight of sludge samples maintained at 40° to 50° C for 24 hr at atmospheric pressure, or for a much shorter time under vacuum. The result thus obtained is in good agreement with that calculated from the acidity decrease of the sludge during such time. These are, however, only laboratory results, and under the actual sludge storage conditions flow measurements of the gas issuing from the sludge tank have so far shown that only about 1 to 1·5 per cent by weight of the sludge is available as SO_2.

Even on this basis, about half the annual make-up requirement (100 tons) of liquid sulphur dioxide for the Edeleanu plant is made available.

Although not strictly a method of sludge utilization, mention may be made in this connexion of a procedure which has been developed for recovering appreciable quantities of SO_2 from the acid oils produced in the white oil plant. In this case the acid oil is flashed into a vacuum (approx 29 inch Hg); the gas thus released has an analysis very similar to that given

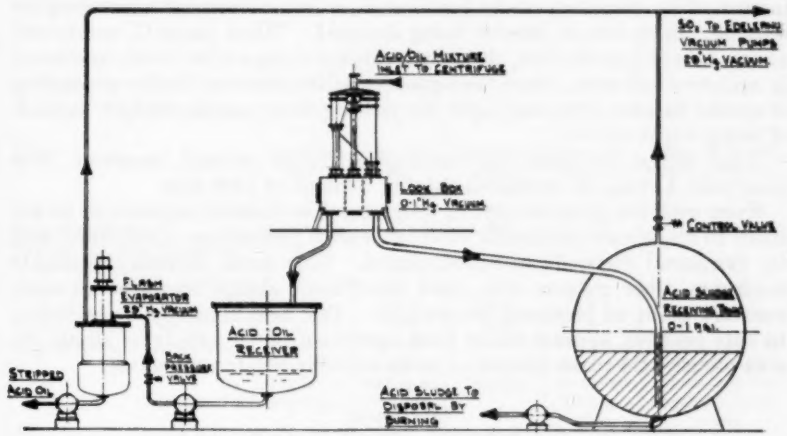

SO₂ TO EDELEANU VACUUM PUMPS 29"Hg VACUUM
ACID/OIL MIXTURE INLET TO CENTRIFUGE
LOCK BOX 0-1"Hg VACUUM
CONTROL VALVE
FLASH EVAPORATOR 29" Hg VACUUM
ACID : OIL RECEIVER
BACK PRESSURE VALVE
STRIPPED ACID OIL
ACID SLUDGE TO DISPOSAL BY BURNING

FIG. 2

above, and has, for some months, been fed to the Edeleanu plant to provide some of the make-up SO_2 required.

Fig. 2 shows the schematic arrangement for SO_2 recovery from sludges and acid oils.

Sulphonic Acids

Oleum sludges from the treatment of mineral oil contain in solution appreciable quantities of sulphonic acids. Whilst these acids thus dissolved in the sludge are of both the " mahogany," or oil-soluble, and the " green," or water-soluble, types, the latter tend to predominate, the former being retained to a greater extent in the acid oil.

At present, in the absence of any pre-treatment of the oleum sludges prior to burning, these acids are destroyed when the sludge is used as fuel. Some of the sulphonic acids present in oleum sludges can, however, be extracted by washing these sludges with mineral oil, and the yield of oil-soluble sulphonic acids, which are normally recovered from the acid oil, is thereby considerably enhanced. However, no major reduction in the sulphur content of the sludges can be expected from this process, since the sulphur content of the recovered sulphonic acids is only of the order of 5 to 10 per cent.

Conclusions

Our future programme for dealing with sludge envisages, in the case of oleum sludges, the more complete recovery of SO_2 and the removal of a proportion of the sulphonic acids that they contain. Both these developments will be based on the procedures which have been outlined above. However, after such treatment, there seems to be little else that can be done with the residual sludges except burn them. Burning will likewise be instituted for disposing of the liquid sludges from industrial lubricating oil refining, which are at present being dumped. When plant C, mentioned above, is in full production, the output of solid sludges from batch treatment in agitators will cease, since the agitators will be reserved for the processing of special batches of mainly light oils yielding fairly mobile sludges, capable of being burnt.

Total sludge available for burning will thus amount to about 7000 tons/year, having an estimated sulphur content of 1300 tons.

Even with the present sulphur shortage, this quantity appears to be too small to make recovery worth while, at least if production of sulphuric acid by the usual methods is contemplated. This same difficulty probably confronts other refiners who have insufficient sludge to make an acid-recovery plant an economic proposition. The most constructive approach to this problem appears to be joint operation of, or supply of sludge to, a sulphuric acid plant by two or more suitably located refineries.

References

[1] Kaye, H., and Spencer, R. K. *Oil Gas J.*, 9.2.50, **48** (40), 80.
[2] American Petroleum Institute, "Disposal of Refinery Wastes," Section II, 2nd Edn. 1938, p. 28.

ACID SLUDGE—ITS UTILIZATION AND DISPOSAL

By E. S. Squire,* F.R.I.C. (Fellow), D. G. Pidgeon,* M.B.E. (Associate Member), and P. C. Jones *

Introduction

Sulphuric acid has been used as a refining agent for petroleum oils since the earliest days of the oil industry and, despite the development of a large number of chemical, physical, and catalytic refining processes, it has remained pre-eminent as a reagent in the manufacture of a very wide range of products.

Under early conditions of small-scale operation, the disposal of the relatively small quantities of spent acid was straightforward, but with the continued growth of the industry and the increasingly exacting requirements of legislation dealing with atmospheric pollution, traditional methods of disposal have become inadequate, so that the modern refinery has been faced with ever-increasing problems in dealing with this troublesome waste material.

* Anglo-Iranian Oil Co. Ltd.

These problems are accentuated by the widely diverse characteristics of the acid tars produced in refining, with the result that no one method of disposal or recovery is universally applicable. Thus, tars may range from the mobile liquids produced from white oil treatments, through the less fluid spindle oil tars, to the highly viscous sludges left after treatment of heavy lubricating oils and petrolata. By comparison with the liquid tars, the latter present most difficulty in handling, since although capable of being pumped when freshly prepared, they change rapidly to an unpumpable solid or semi-solid mass on exposure to air.

TABLE I

Typical Analyses of Acid Tars

Tar	Benzine tar	Kerosine tar	Spindle oil tar	Medium lubricating oil tar	Heavy lubricating oil tar
Specific gravity	1·6	1·4	1·3	1·2	1·2
Titratable acidity as wt % H_2SO_4	55·4	45·8	42·9	18·3	32·7
Free sulphuric acid, wt % H_2SO_4	46·0	33·2	39·7	17·3	—
Water content, wt %	3·3	5·6	7·6	3·8	—
Appearance	Mobile liquid, stable on storage.	Mobile liquid, stable on storage.	Fairly viscous liquid.	Viscous material, but still pumpable.	Extremely viscous material which rapidly ages to solid mass.
Viscosity at 100° F, cs	28·8	331	3410	—	—

Typical analyses of different types of acid tar are given in Table I. The lighter tars consist largely of a mixture of unchanged sulphuric acid and sulphonic acids with smaller amounts of water, oil, and sulphur dioxide. Alkyl and dialkyl sulphates appear to be absent from the tars derived from straight-run products. The nature of lubricating oil tars is less certain, but they contain lower acid contents and more hydrocarbon matter than the liquid tars.

In addition to tars from refining treatments, refineries manufacturing aviation products also produce large quantities of spent acid from alkylation processes. These alkylation discard acids contain some 85 to 92 per cent of sulphuric acid, 6 to 13 per cent of hydrocarbons, and the balance water, and large quantities are re-used in place of fresh acid in refining treatments.

The magnitude of the disposal problem for these materials can best be illustrated by reference to Tables II and III, which show respectively sulphuric acid flowsheets for a large Middle East refinery possessing extensive alkylation capacity with an acid-recovery system, and for a smaller home-based refinery processing lubricating oils and with no recovery.

METHODS OF DISPOSAL BY DESTRUCTION

It is very rarely that acid tars can be dumped without causing a serious nuisance, and if no methods of recovery are available they must be burned. The method of burning depends on the types of tar and the quantity.

TABLE II

Sulphuric Acid Flowsheet for Large M.E. Refinery with Acid-recovery System

All quantities expressed in tons of 98 per cent sulphuric acid

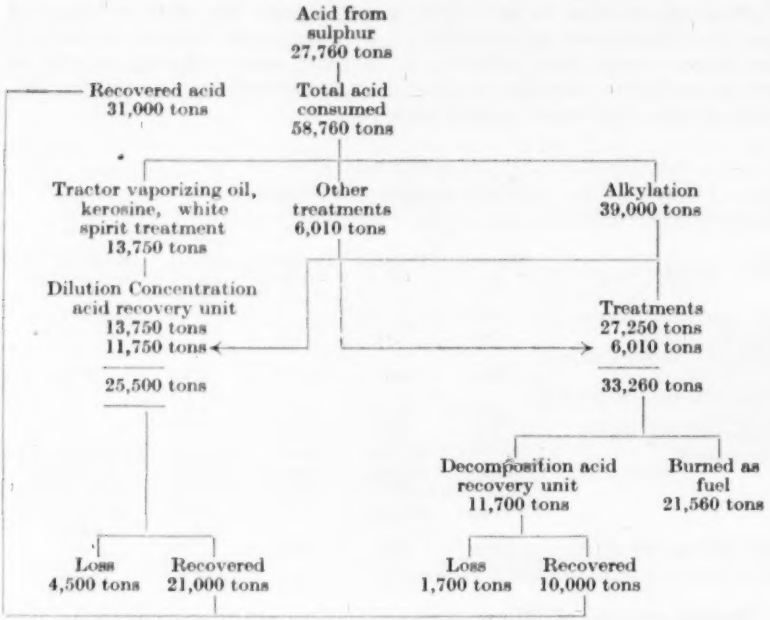
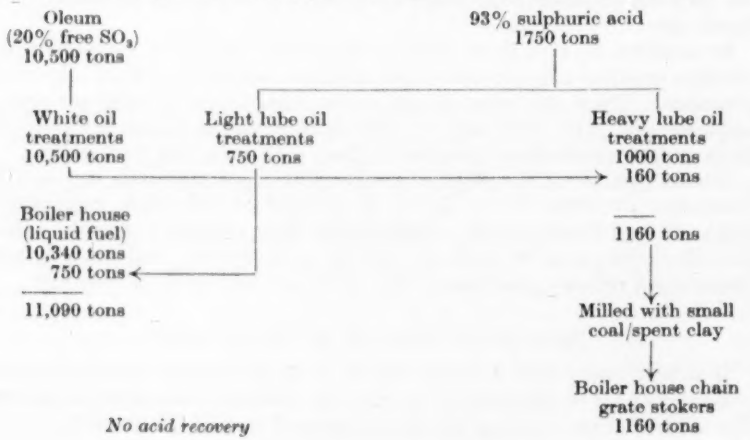


TABLE III

Acid Tar Flowsheet for U.K. Refinery Manufacturing Lubricating Oils

All quantities expressed in tons/annum of oleum and 93 per cent sulphuric acid


Oleum (20% free SO_3) 10,500 tons
93% sulphuric acid 1750 tons
White oil treatments 10,500 tons
Light lube oil treatments 750 tons
Heavy lube oil treatments 1000 tons
160 tons
Boiler house (liquid fuel) 10,340 tons
750 tons
11,090 tons
1160 tons
Milled with small coal/spent clay
Boiler house chain grate stokers 1160 tons
No acid recovery

Liquid Tars

Large quantities of liquid tars are destroyed by burning as liquid fuel under boilers. Customarily one tar burner is used in conjunction with five or six oil or gas burners.

Many types of burner have been tried, but one which has been found to be both simple and effective is an air-operated type consisting of a short length of steel piping, with a screwed-on nozzle having drilled holes around the periphery. The air and tar are fed together through the burner.

Occasional wastage troubles have been encountered due to traces of moisture in the compressed air. Cast iron has been found to be the most satisfactory material for the nozzle, with a life of up to 2 to 3 weeks. Replacements are readily fabricated in the local workshops.

Since a universal type of burner suitable for use with both tar and oil fuel would present many attractions, consideration has also been given to conventional burner equipment. Excessive corrosion tendencies limit the use of steam-atomizing burners, but experience has been gained of the operation of several pressure-type burners on white oil tars. While these have operated successfully for short periods, blockages due to carbonization of the orifices occur. Since mechanical atomization is very dependent on the characteristics of the fuel, day-to-day fluctuations in viscosity, due to the normal variations in refining programmes, are an added difficulty in attaining a steady performance with these burners, and none is yet in regular operational use.

In the case of the more viscous but still liquid tars from the treatment of medium-viscosity lubricating oils, a variation in procedure is adopted. Atomization is not attempted, but the tar is broken up by high-pressure steam and sprayed over the incandescent fuel bed of a chain-grate stoker, which helps to complete the combustion. The design of the burner overcomes the usual difficulties due to the immediate carbonization of lubricating oils when contacted with steam, since the tar does not contact the steam until it leaves the orifice. It is then blown clear by the cross jets of superheated steam.

In general, it must be stated that these processes have operated remarkably well, over many years, to produce large quantities of steam, and that anticipated troubles due to corrosion have been much less than expected. The method is limited to the medium- and large-size installations, however, since the low calorific value of acid tars prevents their satisfactory use in small boilers. In these circumstances destruction in a separate furnace is necessary.

Solid Tars

Tars from the treatment of bright stocks and other heavy lubricating oils can be run into bays on to layers of small coal, allowed to harden, and then hand stoked on to a forced-draught coal-fired hearth.

Another method is to use an edge runner mill in which tars and other refinery waste such as tank settlings are milled with small coal. The product is then burned on chain-grate boilers, sandwiched between alternate truck-loads of coal.

Major objections to this procedure are the frequent alterations in the

proportions of the mix (from 3 to 8 parts of coal being required to 1 part of sludge), made necessary by variations in consistency of the tar, the sweating of oily matter in burning, and the difficulties of aerating such finely ground fuel. Briquetting has been tried as a means of improvement, but the combustion characteristics of the briquettes are little better than those of the original ground product. The best procedure as yet tried is to granulate coarsely a mixture of solidified tar, coal, and spent oily clay through toothed cutters. It is possible to produce a fair output of steam with this fuel, but it is difficult to maintain a good solid fuel bed, and the maintenance required on the grate is increased due to the flow of tar through the links.

It is evident that a chain grate is inherently unsuitable for the combustion of such material except when the proportion of tar to coal is small, and that there is a definite need for a new type of grate, preferably with a solid hearth, without complication of moving parts, to take the solid oily wastes. Alternatively, a new process which would entirely eliminate the necessity for dealing with such materials in this way would be welcomed.

Methods of Disposal Involving Recovery of Useful Products

Recovery processes for acid tar can broadly be classified into two groups, involving hydrolysis or thermal decomposition respectively.

Hydrolysis Methods

One of the earliest methods of disposal for liquid tars was by hydrolysis with hot water, followed by neutralization of the weak acid solution with milk of lime. The separated oil, amounting to some 40 per cent on the original charge, was burned as works fuel. The present-day cost of neutralizing the enormous quantities of acid involved makes this method very expensive; moreover, the process does not dispose of the odour nuisance entirely, and the discharge of the neutral effluent is itself a problem. However, processes have been developed in which the sulphuric acid content is utilized, either for the recovery of concentrated acid or for the manufacture of a further by-product.

The Recovery of Acid by Concentration. A plant is available at Abadan to recover sulphuric acid from acid sludges by dilution to remove organic matter, followed by re-concentration. Since this process is extremely sensitive, considerable development effort has been expended in acquiring a knowledge of the factors governing the choice of the best feedstock.

The plant, which was designed to recover 135 tons/day of sulphuric acid from tar equivalent to 156 tons of acid, consists of four atmospheric hydrolysers (Fig. 1) in which the acid sludge is added batch-wise to sufficient steam condensate to give an aqueous layer of approximately 50 per cent weight acid. Live steam is used to agitate and raise the temperature of the contents to 250° F. After settling of the two layers, the weak acid is run off to intermediate tankage, while the oil layer is diverted to receivers for subsequent burning in a furnace to which the vapours arising from the hydrolysis stage are also conducted. The hydrolysing cycle and weak acid storage is so designed that the feed of weak acid to the concentrators is continuous.

Reduction of the carbon content of the acid layer by atmospheric hydro-

lysis may be insufficient, and further reduction can be effected by pressure-hydrolysing treatment at higher temperatures. Three pressure treaters rated at 120 lb working pressure are provided, but have only rarely been used in practice, since it is preferred to process sludges which permit the direct concentration of dilute acid liquor.

The weak acid then passes to two two-stage drum concentrators (Fig. 2) in parallel for concentration to 92 per cent strength by hot countercurrent flue gases from the burning of fuel gas. The hot gases are supplied to the

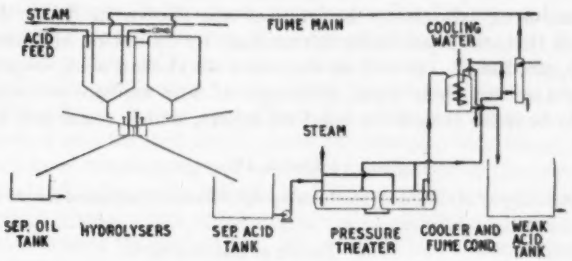


FIG. 1

DILUTION CONCENTRATION ACID RECOVERY PLANT. HYDROLYSING SECTION

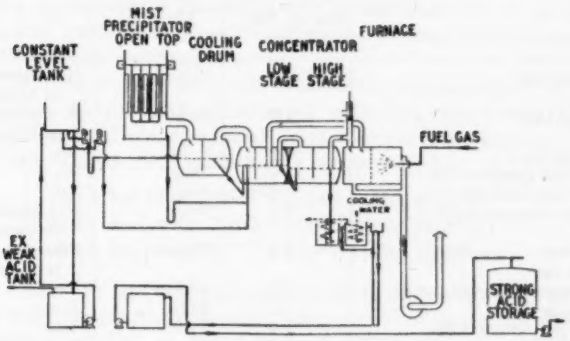


FIG. 2

DILUTION CONCENTRATION ACID RECOVERY PLANT. DRUM CONCENTRATORS

two stages at 1100° F, the gas from the high-concentration stage also passing to the low-concentration stage. The gas flow to the stages is controlled to give an exit temperature from the high stage of not less than 380° F, and from the low stage of not more than 270° F. By operating to these temperatures foaming is avoided. The exit gases pass through a cooler to a mist precipitator for removal of entrained acid before being discharged to the atmosphere. The concentrators, like the dilution tanks and pressure-treatment vessels, are lined with lead and acid-proof brick, and the gas linee are of Duriron.

The concentrated acid from the high stage flows through coolers to storage tanks, from which it is sent for fortification to 98 per cent acid at adjacent sulphur-burning contact acid plants.

Selection of Feedstocks. A suitable sludge for recovery by this process must yield on hydrolysis :—

(i) An acid layer which does not contain so much carbonaceous matter that charring or foaming would become troublesome in the concentrator. A maximum carbon content of 1·5 per cent weight (based on 100 per cent H_2SO_4) in the diluted acid is stipulated; above this figure further reduction by pressure hydrolysis is necessary.

(ii) An oil layer which is fluid and readily pumpable.

The results of laboratory hydrolyses are shown in Table IV, carbon contents of the acid layer being determined by oxidation and measurement of the CO_2 produced. It will be seen that alkylation acid, *iso*pentane, and *iso*hexane tars on their own, although of low carbon content, are unacceptable because they form solid oil layers, which could not be disposed

TABLE IV

Suitability of Acid Tars for Recovery by Dilution/Concentration Process

Acid tar	Result of dilution to 45 wt-% acid		Suitability for recovery
	Acid phase: Wt-% carbon on 100% H_2SO_4	Oil phase: Fluidity	
*iso*Pentane tar *	1·3	Solid	Not alone, acceptable in blend †
*iso*Hexane tar *	1·7	Solid	Not alone, acceptable in blend †
*iso*Heptane tar *	4·6	Solid	No
Straight-run gasoline tar	16·7	Solid	No
Pressure distillate tar	6·5	Solid	No
Alkylation discard acid	0·2	Solid	Not alone, acceptable in blend *
Straight-run gasoline SO_2 extract tar	5·0	Fluid	Pressure hydrolysis required
Tractor vaporizing oil tar	1·0	Fluid	Yes
Kerosine tar	1·1	Fluid	Yes
Kerosine SO_2 extract tar	1·3	Fluid	Yes
White spirit tar	2·1	Fluid	Probably yes
Composite tar	10·4	Semi-solid	No

* From the refining of superfractionator products.
† Blend must be capable of producing liquid oil phase.

of from the hydrolysers. *iso*Heptane, straight-run gasoline, pressure distillate, and composite refinery tars are unsuitable because they produce both high carbon content acid and solid oil, while straight-run gasoline SO_2 extract tar, although giving a fluid oily layer, produces acid with a high carbon content, which would require additional pressure treatment. Kerosine, tractor vaporizing oil, and probably white spirit tars only are capable of being separately processed. It has been found possible, however, to extend the range of possible feedstocks by the incorporation with these tars of alkylation acid or *iso*pentane and *iso*hexane tars in proportions such that the oily layer from the hydrolysed blend remains fluid.

High-acidity alkylation discard acid can neither be burned in a steam battery nor processed in decomposition-type plants, while the *iso*paraffin tars, as will be seen later, are quite suitable for the latter process. The best feed is a blend of kerosine and white spirit tars with the maximum permissible proportion of alkylation acid. This has the additional advantage that the quantities of oil produced for disposal are a minimum. In practice, blends containing up to 70 per cent of alkylation discard acid have been hydrolysed successfully.

Although such a scheme, involving segregation and a dual feedstock, has certain disadvantages, it has been found successful in operation, and has steadily produced up to 2500 tons of recovered acid per month. Experience has shown that the acid tars and discard acid should be processed immediately after production, since even twenty-four hours' storage can result in difficulties during hydrolysis due to the formation of sludge. Introduction of acid tar into the condensate must also be gradual, otherwise the resultant local overheating can cause polymerization of the unreacted tar.

Mechanical difficulties encountered with the plant have been those usually encountered in plants where extensive use is made of lead-lined equipment operated at elevated temperatures. No troubles have been experienced in connexion with the drum concentrators, except with occasional choking of the acid-transfer lines and cracking of some of the Duriron pipes.

Use of Dilute Acid for the Manufacture of Ammonium Sulphate. At Pumpherston advantage is taken of the availability of cheap ammonia liquor from the washing of shale retort gases to manufacture ammonium sulphate as a saleable by-product.

Acid tar, derived mainly from the treatment of shale diesel oil, is given three successive 25 per cent treatments with hot water, the first wash liquor being the third-stage effluent from the previous batch. The acid strength of the effluent from each wash is approximately as follows :—

from 1st wash 50° Twaddell (34% H_2SO_4)
from 2nd wash 35° Twaddell (24% H_2SO_4)
from 3rd wash 20° Twaddell (14% H_2SO_4)

The first two washings are combined to form the weak acid feed to the ammonium sulphate plant, fresh acid being added at this stage to bring the concentration up to 36° to 40° Twaddell (25 to 30 per cent H_2SO_4) if necessary.

Dilute ammonia liquor from the shale-oil works is stripped by steam in a mild-steel stripping column, lime being added to complete the process and the ammonia vapour passed direct to lead-lined reaction vessels, where it is absorbed in the weak acid until a faint smell of ammonia from a breather pipe indicates that reaction is complete. After separation of nitrogen-base tars and filtration, the ammonium sulphate liquor then passes to conventional evaporator equipment for the production of crystals, which are water washed in basket-type centrifuges and finally air dried in a rotary drier at 160° F.

Some 20 tons of ammonium sulphate of Grade I specification are produced in this manner daily, but since labour and operating costs are high, the process is likely to be attractive only where the disposal of large quantities of dilute ammoniacal liquor is also a problem.

Other Uses of Dilute Acid. White oil tars have also been investigated as a possible starting point for the production of surface-active materials and of dilute acid liquor for de-rusting and pickling operations. The separated oils from certain tars have also been found to have potentialities as acid corrosion inhibitors and wood preservatives, but none of these developments would appear to offer sufficient scope as a major outlet for present-day quantities of acid tar.

Acid Tar Decomposition

This process is designed to recover sulphuric acid from acid tars by thermal decomposition, in contact with hot coke, to produce sulphur dioxide which is passed to a contact acid plant.

The plant at Abadan was built to process a maximum of 123 tons/day of acid tar of the specification given in Table V, with a minimum of 10 tons/day

TABLE V

Decomposition Assay of Acid Tars

Acid tar	Weight per cent						Per cent recovery	Suitability for recovery alone
	Coke	Oil	Water	SO_3	Equiv. H_2SO_4	Titratable H_2SO_4		
Design feed	32·7	1·7	25·3	40	—	—		
Alkylation discard acid	51·5 *	Nil	13·4	31·5	48·5	92·9	52·2	No
isoPentane tar †	9·8	Nil	27·7	53·4	81·3	86·8	94·0	No
isoHexane tar †	12·2	Nil	31·6	50·1	78·1	80·6	97·0	No
isoHeptane tar †	16·7	Nil	31·4	46·3	70·9	73·8	96·0	Yes
Straight-run gasoline tar	22·4	6·7	29·3	34·5	52·8	58·8	90·0	Yes
Straight-run gasoline tar	24·8	1·7	27·4	41·2	63·1	63·7	99·4	Yes
Pressure distillate tar	33·3	11·8	26·4	22·5	34·5	42·0	82·0	Yes
Straight-run gasoline SO_3 extract tar	23·2	12·2	24·3	36·4	55·8	56·2	99·4	Yes
Kerosine tar	29·0	15·2	23·5	—	35·5	40·2	88·4	Yes
Tractor vaporizing oil tar	—	12·7	—	—	—	58·8	—	Yes
Typical composite tar	24·4	9·3	32·0	32·3	49·5	56·2	88	Yes
Composite tar + 24% alkylation discard acid	19·9	4·3	31·1	41·3	63·3	66·4	95	Yes

* "Coke" contained 89% H_2SO_4 by weight.
† From the refining of superfractionator products.

of raw sulphur. Ninety-five tons/day of 98 per cent acid were to be produced at a recovery efficiency of 86 per cent on sulphur dioxide evolved from acid tar and 90 per cent from sulphur burning.

In the plant (Fig. 3), coke is preheated in a rotary kiln by means of fuel gas, to a temperature of 900° to 1100° F, and is then passed into a smaller rotary kiln into which the acid rar is injected. Decomposition of the tar occurs into SO_2, water, hydrocarbons, and more coke, which is returned to the heating kiln via a series of elevators, conveyors, and storage bins.

The decomposition gases then enter the primary purification section, which consists of a scrubbing tower and two water-cooled condensers in series. A circulating flow of water over the scrubbing tower removes entrained coke dust and effects a partial condensation of oil and water vapour, and the cooled gases leave the condensers at 110° to 130° F.

The gases are then picked up by a blower and discharged through a cyclone separator and catchpot into the SO_2 furnace, where any non-

condensible hydrocarbons are removed by combustion at 1800° to 2000° F. Sulphur can be burned in this furnace to augment acid production if desired, but gas burners are also provided in case the furnace temperature is insufficient when operating on sludge alone.

The products are cooled in a second spray tower, passed through a Cottrell mist precipitator, and are then diluted with air to 7 per cent sulphur

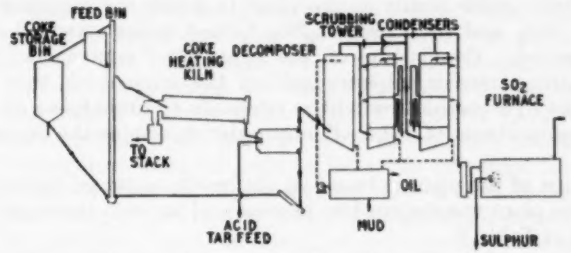


FIG. 3

DECOMPOSITION TYPE ACID RECOVERY PLANT. DECOMPOSER AND PRIMARY PURIFICATION SECTION

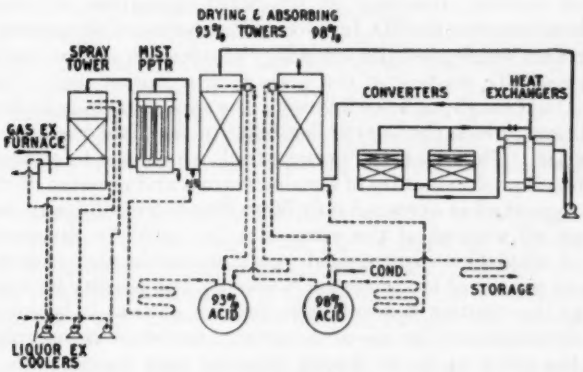


FIG. 4

DECOMPOSITION TYPE ACID RECOVERY PLANT. GAS PURIFICATION AND CONTACT SECTION

dioxide before entering the contact section (Fig. 4). This is of entirely conventional design, comprising a 93 per cent acid drying column, two vanadium pentoxide contactors in parallel, and an SO_3-absorbing tower discharging 98 per cent acid to finished storage.

The main problems to date have been the inability to attain design throughout, coupled with a low time efficiency in the decomposition section. Both have been bound up with the necessity of having to process a feedstock departing widely from the design basis. This has been brought about by the major changes in refining operations resulting from rapid wartime expansion, so that the present tar composition bears little relationship to that obtaining when the plant was originally projected.

To be suitable for recovery by decomposition, an acid tar must contain sufficient hydrocarbon to be able to reduce all the sulphuric acid to sulphur dioxide, with the production of adequate coke to render the process self-sufficient. At the same time the amount of oil and water formed should be a minimum to reduce the load on the purification equipment.

Tars are assessed by a well-known laboratory method simulating the decomposition which occurs on the plant in which the quantities of coke, oil, water, SO_2, and equivalent H_2SO_4 formed under standard conditions are determined. Comparison of the figure for "equivalent" sulphuric acid with the titratable sulphuric acid of the original tar then indicates whether the hydrocarbon content is adequate to decompose all the acid, while the proportions of coke, oil, and water determine the degree of suitability.

The design of the plant is based on the results obtained in this analysis. The Abadan plant was designed to process acid tar with the analysis shown at the top of Table V.

In any given plant the ratio of water to SO_2 and the oil produced will determine the input as the condensing capacity is fixed. It will be seen from Table V that the composite tar which is fed to the plant has a much higher oil yield and water/SO_2 ratio. This has overloaded the primary gas purification section, resulting in untoward quantities of uncondensed hydrocarbons entering the SO_2 furnace. The increased air required to offset this has in turn overloaded the secondary purification section and increased the water and CO_2 content of the gases to the contact unit. Substantial limitations in throughput have followed, due almost entirely to the changed feed conditions, which the limited flexibility of the plant has been unable to accommodate. Thus with the present feed, which due to current refining trends consists predominantly of benzine extract and pressure distillate tars, the plant capacity has averaged only 50 to 60 per cent of design capacity.

The high oil content of the composite tar could be decreased by the addition of alkylation discard acid, and this would also permit a higher yield of acid per ton of tar processed (Table V), but priority for this material is given to the dilution/concentration process so that it is not available. In these circumstances the use of the present feedstock is unavoidable, and to bring the plant up to its design capacity very considerable additions would be necessary to the primary and the secondary purification sections of the equipment.

Poor time efficiencies of only 40 to 50 per cent in the decomposer have largely been the result of mechanical troubles with the coke-circulation system and the difficulties in measuring the coke temperature and the circulation rate. The coke-outlet temperature must be maintained at not less than 500° F, otherwise undecomposed acid can give rise to serious corrosion on the conveyor system. This section of the plant has now been virtually rebuilt, and recent time efficiencies of better than 80 per cent have been recorded.

It has been found that a coke figure of 16 per cent is about the lowest practicable limit to maintain a net balance in coke production, and it is preferable to produce a small surplus. With some of the newer types of decomposer, employing indirect heating, the lower coke limit would be much less, since none is burned.

Conclusions

Sufficient has been said to indicate the problems involved in acid tar disposal, whether by recovery or non-recovery methods. As far as the authors know, no single recovery process will handle all the tars which may be produced in a modern refinery. Whereas thermal decomposition is of wider application than the dilution/concentration method, unless the plant is designed on a very broad and therefore costly basis, the input will vary very considerably with the composition of the tar available. The limitations of contemporary designs are amply illustrated in the case of the Abadan plant, where changes in the type of feedstock have resulted in throughput restrictions which require capital expenditure to remedy.

It will be seen from what has been said that the processes described can be operated only in a large-scale refinery, and there is little likelihood of the smaller refiner being able to recover acid until a method is developed suited to deal with quantities of about 20 tons/day of diverse tars (containing 10 to 12 tons H_2SO_4). Unless recovery of the acid tar output from a number of refineries could be arranged, at present there seems little real alternative to disposal by destruction. Even where recovery units are installed, standby destructor equipment must be provided as an insurance against refining shutdowns due to bottlenecks in tar processing.

There is therefore a definite need for a reliable destruction process. While in the past the burden of tar destruction has fallen on the boiler-house, too much emphasis should not be placed on heat utilization. Although apparent savings in fuel can be made, the net result of saddling a modern steam-raising plant with the requirements of complete acid tar destruction may well be to compromise the efficient working of both processes.

Simple tar burneries are to be preferred, capable of discharging a clean combustion product to the atmosphere, and of sufficient flexibility to take care of all the possible fluctuations in quality and quantity of acid tars. While high combustion efficiency is required, elaborate burners should be avoided, and the aim should be to provide robust and straightforward equipment.

Such an installation may be considered to consist of a generously proportioned combustion chamber where the tars are fired against a mass of incandescent brickwork through air-operated burners of the type already described. The brickwork should be preheated by oil or gas fuel prior to the introduction of acid tar, and complete combustion should be ensured by operating at a furnace temperature of 2000° F with 100 to 200 per cent excess air.

Although a maximum heat release of 20,000 B.Th.U/cu. ft/hr has been suggested for this duty, the much lower figure of 7000 to 10,000 B.Th.U/cu. ft/hr is preferred. Since the stack temperatures must be kept below 1100° F to prevent reconversion of SO_2 to SO_3, the combustion products are cooled to 1000° F by dilution with air through doors fitted to the end of the furnace. Since large quantities of air, of the order of the original volume of gases, are involved, allowance must be made in chimney sizing.

The actual height of chimney is dictated by the daily volume of sulphur dioxide to be discharged, and in hilly country the " effective " stack height

must be taken from some fixed reference level rather than from the ground, to allow for the surrounding topography.

It has been suggested that viscous lubricating oil tars be burned by extruding the freshly prepared material into a sloping-hearth combustion chamber operating under similar conditions to the above. This involves immediate disposal of the sludge before solidification occurs, and therefore requires the plant to be adjacent to the treatment units.

Recent laboratory work, on the other hand, has shown that even highly viscous sludges can be dissolved in liquid white oil tars by stirring at a moderate temperature, provided that they have not been allowed to contact the air; the resultant solutions are both fluid and stable on storage at ambient U.K. temperatures. Characteristics of some of these blends are given in Table VI, and it would appear that further work on these lines

TABLE VI

Properties of Blends of Lubricating Oil Tars with White Oil Tars

Blend	Composition	Temperature for mixing, °F	Pour point, °F	Kinematic viscosity, cs			Decomposition assay					
				100° F	70° F	40° F	Coke	Oil	Water	SO_3	Equiv H_2SO_4	Titr H_2SO_4
A	White oil tar base only	60	<5	243	750	2968	—	—	—	—	—	—
B	White oil tar base + 7% light lube oil tar	60	<5	326	1072	5286	32·6	10·1	23·1	21·1	32·3	43·5
C	White oil tar base + 8% heavy lube oil tar	130	<5	371	1200	5700	—	—	—	—	—	—
D	White oil tar base + 7% light lube oil tar + 8% heavy lube oil tar	120	<5	395	1297	6500	32·3	10·0	23·1	24·4	37·4	44·7

might show promise in unifying the disposal of all types of tar. The decomposition assays of these blends are of interest, since although not ideal for processing on a decomposition-type unit, it would appear that such a process is feasible, provided that adequate quantities of white oil tars are available.

The problem of disposal or recovery of acid tars is a very difficult one, and considerable development effort is being applied to find alternative refining methods which do not result in their production.

ACID SLUDGE—ITS UTILIZATION AND DISPOSAL

By W. E. Nieuwenhuis *

Summary

The utilization and disposal of oil-refinery acid sludge may be effected in three different ways :—

(*a*) utilizing its heating value by burning under a boiler;

(*b*) hydrolysing the sludge and recovering the separated oil and concentrating the resulting dilute sulphuric acid;

(*c*) decomposing the sludge thermally to form SO_2 and coke (reduction at 250° to 300° C) or SO_2 and carbon dioxide (combustion at 1100° to 1200° C), and, after purification, reconverting the SO_2 into sulphuric acid in a contact plant.

From a point of view of utilization, acid sludge has a potential value as a source of sulphuric acid and as a fuel, according to the relative contents of the components. Next to the chemical composition the physical properties of the sludge are factors in determining the applicability of the conversion process. Some types of sludge have a pronounced tendency to polymerize, to harden, or to coke, thus causing considerable handling difficulties. Heavy lubricating oil sludge could hitherto be disposed of satisfactorily only by burning it in a rotary drum-type kiln, less viscous sludges rich in organic material could be burned in gun-type burners after homogenizing the sludge or sludge/fuel oil mixture in a mill or using gas oil as a carrier. Hydrolysis of sludge may be considered for sludges containing 40 to 70 per cent wt sulphuric acid, using Mantius vacuum or Chemico drum-type concentrators to recover the sulphuric acid. Concentration may be omitted, when the dilute acid can be directly employed for fertilizer manufacture (superphosphate and ammonium sulphate). Acid losses in the combined hydrolysis–concentration process may be high, due to acid decomposition to form SO_2 during concentration. In addition, foaming and severe fouling by precipitating organic material may be encountered.

Commercial decomposing plants were developed by Monsanto and Chemico. The Monsanto–Ross system handles spent alkylation acid, producing by-product coke containing 7 to 8 per cent wt sulphur. The Monsanto–Mannheim decomposer is intended to handle not only spent alkylation acid but also sludges richer in organic material.

Chemico's processes are for decomposing either spent alkylation acid at 1100° to 1200° C in a burner, or for processing heavier sludges with a sulphuric acid content down to 50 per cent wt in a rotating coke kiln at 280° C.

Introduction

Although there is a general trend in petroleum refining to reduce the use of sulphuric acid, it is still consumed in large quantities, *e.g.*, in the alkylation process as a catalyst, and in the treating of white products (including gasolines), gas oil, fuel oil, lubricating oils, transformer oil, medicinal white oils, etc. In all instances a spent acid is produced having physical and chemical properties entirely dependent on the type of product treated. The disposal of "acid sludge," as the spent acid is called, has constituted a refinery problem for many years, one of the difficulties at some locations being to comply with regulations that prohibit the discharge of acid into sewers and of acid fumes into the atmosphere. Acid sludge is a black, evil-smelling mixture of indefinite composition and varying viscosity, containing principally free sulphuric acid, sulphonation products, hydrocarbons, and water. In some types of sludge appreciable amounts of high

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polymerization products and coke are found. The sulphuric acid content of acid sludge varies from about 90 per cent wt in spent alkylation acid to 25 to 30 per cent wt in heavy lubricating oil sludge. The content of organic material increases with decreasing sulphuric acid content, and is 6 to 10 per cent wt in the case of alkylation acid, reaching 50 per cent or more in heavy sludges. Thus, acid sludge may be valuable on account of its sulphuric acid content or as a product having a heating value proportional to its fuel content.

The primary objective of converting acid sludge may be either sulphuric acid or fuel oil recovery for economic reasons, or the destruction of an objectionable waste product which cannot be disposed of as such. During the last few years sulphuric acid recovery from spent acids has not only gained importance from an economic point of view, but also, having regard to the increasing raw materials shortage, as a means of reducing the net acid consumption in the petroleum industry.

Hitherto the following methods for disposing of or processing acid sludge have been developed :—

Burning the sludge directly under a boiler.

Hydrolysing the sludge and recovering a more or less neutral fuel oil and a dilute " black acid " which may be re-concentrated.

Decomposing the sludge at high temperatures and recovering the liberated SO_2 for re-conversion into sulphuric acid in a contact plant.

It depends on the type of sludge which of the above methods can be applied. A special class of sludge is produced in the oleum treatment of transformer oil and technical and medicinal white oils, the sludge being rich in valuable sulphonates, which may be recovered by extraction.[1]

Properties of Sludge

As the composition of acid sludges varies widely it is not possible to give systematic data on composition and properties of the different types. However, some generalities may be expressed.

In many refineries no pure acid is used for the various treating purposes, but spent acid produced in one process is re-used in the next. For instance, spent alkylation acid may be employed for treating gasoline, whilst oleum can be used " countercurrently " by treating successively white oil, light machine oil, or then heavier lubricating oils. Spent alkylation acid is very rich is sulphuric acid and poor in hydrocarbons, and is therefore a suitable feed stock for recovery processes (*c*). This type of acid is easily pumped, and prolonged storage presents no difficulties. Increasing difficulties are experienced, however, the richer the sludges are in organic material. Particularly, lubricating-oil sludges must be considered as presenting a separate problem, mainly in view of their difficult handling. Machine oils with a viscosity up to 50 cs still give pumpable sludges, but the sludges from heavier oils tend to coke and harden very rapidly after their production. It has been observed that contact with air strongly accelerates this phenomenon. Transportation and storage of this type of sludge is therefore a serious obstacle to their processing.

Burning of Acid Sludge

For the disposal of sludge, rich in organic material, burning the product under a boiler may seem to be the most direct and easiest way. The main problem, however, is how to transport and to feed the material to the burner. Also the operation of the burner presents difficulties, mainly due to rapid corrosion. The burning of light and medium acid sludges has been practised in several American refineries, and in those European refineries where no objections existed to increasing the amount of SO_2 vented into the air through the refinery stacks.

Thornton [2] reports that a fuel oil/acid sludge mixture was burned under boilers in an American refinery using a burner similar to the familiar heavy fuel oil burner of the steam-atomizing type, and inserted in the Peabody gas-

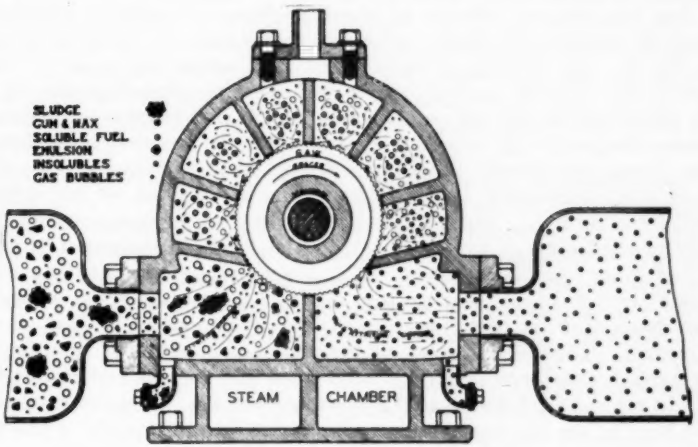

Fig. 1

VOLCANO ACID SLUDGE MILL

ring burners with which the boilers were equipped. One of the difficulties experienced was that the components tended to separate, causing the burners to receive acid sludge, and oil alternately, with consequent irregular burning. Improvement was obtained by homogenizing the acid sludge in a vertical-type mill. This device consists of a vertical cylindrical shell of steel containing a central shaft on which are mounted a number of solid toothed wheels. The shaft is driven by a 50-h.p. motor. Approximately 12 barrels/hour of sludge were passed through the mill in the unit described.[1]

The same company also manufactures mills with a horizontal shaft (see Fig. 1). Other refineries report that the sludge mills do a reasonably good job in blending acid sludge with fuel oil to make a flux for burning. Some types of sludge contain a high percentage of coke, and their heterogeneous structure make them unsuitable for being fed to gun-type burners. The mill acts as a grinder to reduce particles in the mixture to a size which does not plug burners. However, their maintenance is excessively high,

the rotors requiring replacement about every four to five months. It was also reported that the mills did not entirely prevent separation of the sludge from the fuel oil in the boiler-house fuel tanks.

A different method of pumping heavy acid sludge is a circulating gas oil " carrier " system, whereby acid sludges are injected into a circulating stream of gas oil which carries the sludge in suspension into a settling tank at the boiler-house. The acid sludge is settled out of the gas oil and pumped from the cone-bottomed settling tank to the burners. The gas oil layer overflows from the top of the settling tank into a carrier surge tank, from which it is recirculated. A disadvantage of the system is the inevitable loss of some gas oil through entrainment in the sludge fed to the burners, thus degrading a certain amount of valuable gas oil to refinery fuel. Plugging of lines by hardened acid sludge has also been experienced.

A less troublesome method of burning heavy acid sludge consists of feeding the sludge immediately after its production to a rotary drum-type kiln, the flue gas of which is passed through a water-tube boiler for heat recovery and then to a stack. It is necessary to emphasize that in all cases where acid sludge is being burned under boilers there is an increased corrosion danger for the economizers, and it may be better to omit these.

The drum-type burner consists of a steel shell, internally lined with acid-resisting refractory bricks. The drum is installed in an inclined position and rotated. The acid sludge is fed by gravity continuously into the drum and ignites on the brick-work, which has been preheated by burning fuel in an auxiliary burner. The lower end of the rotating drum extends with a small clearing into a fixed after-combustion chamber, where residual coke particles, formed by decomposition of the sludge, are burned. The effluent gases of the combustion chamber then further pass through a water-tube boiler. In a drum of 6 feet dia and 20 feet long 12 to 14 tons of acid sludge could be burned per day. One essential point to be kept in mind in installing an acid-sludge burning system is to locate the drum burner as closely as possible to the sludge discharge at the treating plant. When plotting refinery extensions it is a good policy to build the treating plant at the point where facilities for the removal of acid sludge can best be located, since pumping of lubricating oils over a large distance is no problem and the transportation of acid sludge should be avoided. Excluding sludge from contact with air is also a condition for preventing handling difficulties. It is a well-known fact that centrifuge acid sludge is less viscous than agitator acid sludge.

Hydrolysis and Re-concentration

When treated with a sufficient weight of water, acid sludge separates into two layers : an upper tarry layer rich in organic matter and a lower aqueous layer containing most of the spent sulphuric acid and a weak solution of highly aromatic disulphonic acids. The upper layer contains neutral oil and sulphonic acids. By heating the acid sludge/water mixture and adding a hydrocarbon oil for dissolving the organic material, the separation of the sludge into a dilute acid and an oil layer which can be utilized as a refinery fuel is facilitated. The hydrolysis is carried out in a cylindrical, cone-bottomed, lead-lined sludge kettle. A temperature of

approximately 220° F is attained by open steam coils, and the charge is agitated for a period. It is then allowed to stand until the sludge and dilute acid form separate layers. The organic layer is pumped to other cylindrical, lead-lined, cone-bottomed tanks, where further acid separation is effected by additional agitation. The acid is again separated and the organic sludge is delivered to the boilers as fuel. This method has been practised in many refineries for the disposal of light acid sludges (*e.g.*, those produced in gasoline and kerosine acid treatment) only to save fuel; the dilute acid solution was discarded. The latter procedure, however, creates a new disposal problem, and may not be tolerated at many locations. In some cases the dilute acid could be sold to superphosphate manufacturers or could be converted into ammonium sulphate fertilizer. However simple the method of hydrolysis may seem, considerable difficulty is encountered because of emulsions, frothing, sludge solidification, etc.[3]

If sulphuric acid recovery is also desired, the weak acid layer, ordinarily having a strength of 30 to 55 per cent wt H_2SO_4, can be concentrated. In many cases serious difficulties are encountered and even in the most modern equipment it is impossible to concentrate sludge acid above 92 to 93 per cent wt.

The weak acid always contains a certain amount of organic material, varying from 2 to 4 per cent C calculated on the amount of H_2SO_4 present. During the concentration, 40 to 60 per cent of the carbon precipitates in the acid as soon as the acid concentration reaches a value of 60 to 75 per cent. Above that concentration the organic materials decompose acid with formation of sulphur dioxide, which has to be vented, causing an economic loss in addition to the liberation of objectionable gas. The reduction of sulphuric acid by organic material is primarily due to the hydrogen atoms of the hydrocarbons and not to the carbon atoms. As the hydrolysis acid may contain up to 1·5 per cent hydrocarbons and these can destroy up to as much as nineteen times their weight of sulphuric acid, it is possible for acid losses to rise as high as 30 per cent. Usually the acid is concentrated in two or more stages in order to avoid foaming and to remove separated carbonaceous material. Air blowing during concentration may sometimes be helpful in overcoming these difficulties. The form in which the organic material precipitates depends on the origin of the sludge, that is to say of the type of oil treated and the type of acid used.

Sludges from naphtha and kerosine give a carbon precipitate which remains dispersed in the acid handled in the concentrators but separates easily in the settling vessels installed before the last concentration stage. The floating coke is removed from these settlers. Sometimes the organic materials form a pasty layer floating on the acid in the concentrators. This layer cannot be removed during operation and is rapidly converted into coke, necessitating cleaning the equipment after 2 to 7 days, sometimes even after 12 hours.

Spent alkylation acid may give rise to the formation in all the concentration equipment of a very viscous, even gelatinized, acid, but only if the spent acid has not been sufficiently diluted before being fed to the concentrators. The spent acid should be diluted to 25° to 40° Bé, and at least 10 per cent of hydrolysed acid sludge ex-gasoline treating should be added.

The concentrated sludge acid of 86 to 88 per cent strength still contains 3 to 4 per cent organic material, and is called black acid. Finely dispersed carbon may be removed by filtration through glass wool, or the organic material may be oxidized by addition of nitric acid before the last concentration stage, thus producing a white acid.

There are various types of commercial concentrators described in the literature.[4-6] The *Mantius* evaporator-type re-concentrator works under vacuum in order to lower the working temperature. The evaporator chamber consists of a steel vessel lined with lead and acid-resistant bricks.

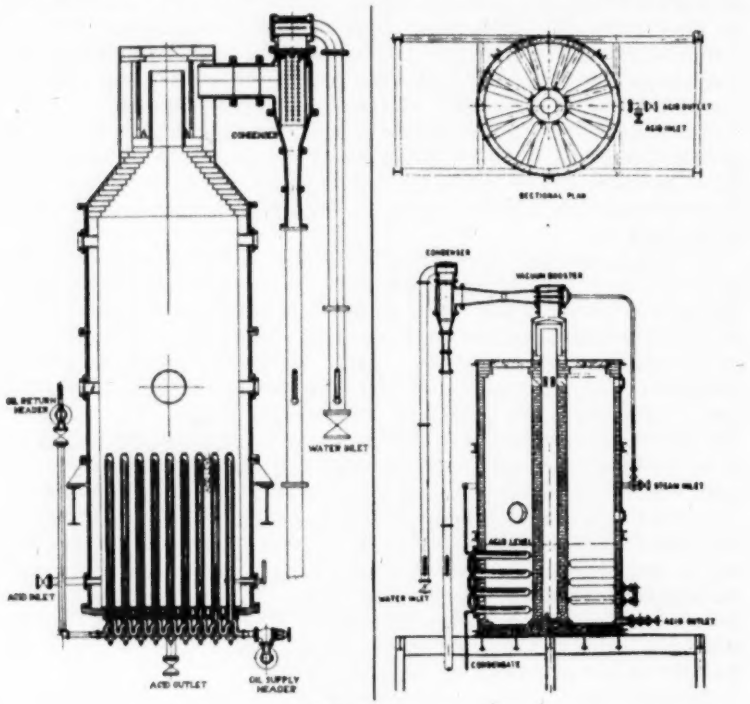

FIG. 2 (*a*)

MANTIUS SULPHURIC ACID CONCENTRATORS (*Chem. Engng Prog.*, May 1947)

For the heating elements the following materials are used, dependent on the concentration range of the acid: 14 per cent silicon iron, Hastelloy D, Karbate, St Joe chemical lead + 6 per cent antimony, tantalum. Vacuum is obtained with steam jets built of special material. The distilled vapours are condensed in barometric condensers with direct water injection. Some types of commercial *Mantius* concentrators are shown in Fig. 2.

In the *Chemico* drum-type concentrator the low temperature of concentration is obtained by passing a large amount of hot gas through the liquid, thus effecting evaporation at a low water-vapour partial pressure (Fig. 3). It is essential that the effluent gases from the evaporator be passed

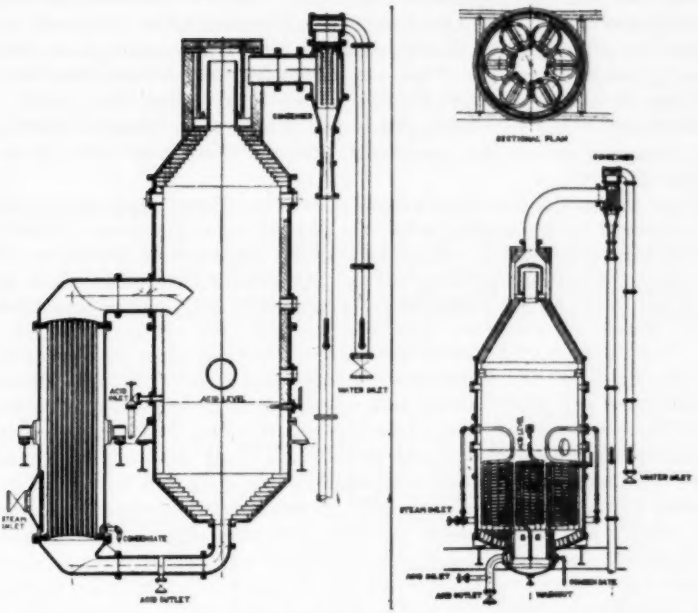

FIG. 2 (*b*)

MANTIUS SULPHURIC ACID CONCENTRATORS (*Chem. Engng Prog.*, May 1947)

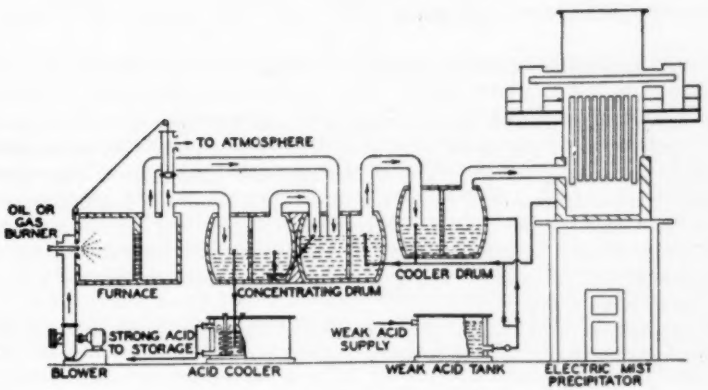

FIG. 3

"CHEMICO" DRUM-TYPE SULPHURIC ACID CONCENTRATOR
(*Chem. Engng Prog.*, May 1947)

through an acid-mist precipitator in order to remove the entrained droplets of sulphuric acid. In the latest type of concentrator, fuel and air are burned in a submerged burner, and the hot combustion gas is released directly under the surface of the acid, where it gives up most of its heat and emerges at a temperature slightly above that of the acid itself. The combustion of the fuel takes place in the dip pipe, which withstands a high temperature on the inside and corrosive action of the acid on the outside (Fig. 4).

Since, in the Mantius-type concentrator, the effluent gases are condensed and removed by the cooling water, no objectionable gases are released into the atmosphere. Acidic water has to be disposed of, however. With the Chemico-type evaporator, on the other hand, an acidic stack gas is produced, but no liquid effluent pollution problem has to be considered.

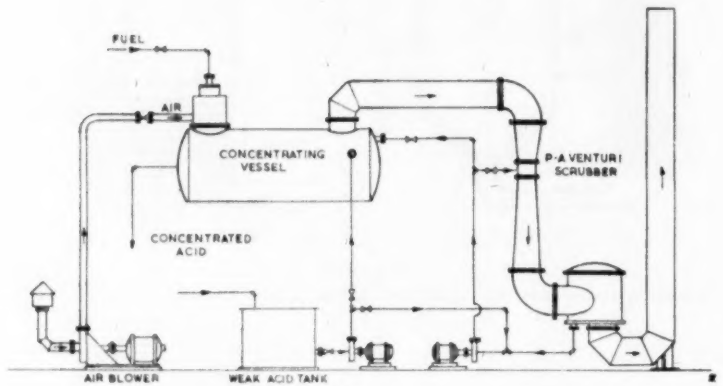

FIG. 4

"CHEMICO" SUBMERGED BURNER SULPHURIC ACID CONCENTRATOR

A third concentration method was proposed by Kalous [14] in which the combustion gas in the drum-type evaporators is replaced by superheated steam. It is claimed that less difficulties are encountered due to the organic material present in the dilute acid and less acid is decomposed into SO_2. In addition, the sulphur dioxide liberated during the concentration may be recovered in gaseous state by condensing the vapours leaving the concentrators; practically SO_2-free water condensate drains off and wet SO_2 escapes as a gas.

If considered worthwhile, the SO_2 after it has been dried, may be converted into sulphuric acid in a contact plant.

DECOMPOSING ACID SLUDGE

This method, in which the acid is broken down to form SO_2, is entirely directed towards sulphuric acid recovery from the spent acid, and therefore does not lend itself for processing sludges rich in oil. The decomposition of the acid may be affected in two different ways :—

(*a*) by heating the sludge at moderate temperatures (250° to 300° C), whereby the sulphuric acid is reduced to SO_2 by chemical reaction with the organic material present, which is converted into coke, practically no CO_2 being formed;

(*b*) by complete combustion of the organic material and thermal decomposition of the sulphuric acid into sulphur dioxide and water at high temperature (1100° to 1200° C).

Low-temperature Reduction

When heating acid sludge containing organic material at temperatures above 250° C, the acid enters into reaction with the hydrogen atoms of the organic compounds to form sulphur dioxide and water vapour.[7, 8 (p. 644)] At the same time cracking of the organic material takes place yielding gaseous and condensable hydrocarbons. The residue is a coke containing 7 to 8 per cent sulphur. Approximately 80 to 85 per cent of the sulphuric acid present in the sludge may be recovered, the remainder of the sulphur being lost partly in the coke and partly, during the purification of the SO_2, in the wash water and condensate. After cooling the produced SO_2 with water in order to condense the hydrocarbon oils and water vapour, the gaseous hydrocarbons are burnt in a furnace, heated by burning refinery gas or better hydrogen sulphide or sulphur. The latter procedure compensates for the overall refinery acid losses and produces the amount of sulphuric acid required for the various processes. The overall acid loss consists of mechanical losses during handling, loss through acid entrainment in the treated products, acid discarded with sludge unsuitable as a feed stock for the sulphuric acid recovery plant, sulphur loss in the decomposer unit, and finally conversion loss in the contact plant.

The SO_2 leaving the combustion furnace is cooled, dried, passed through a Cottrell mist precipitator, and is then fed to a contact sulphuric acid plant.

The low-temperature sludge-decomposing plant as described above is marketed in two different versions: the *Chemico* and the *Monsanto* design.

In the *Chemico* commercial unit the sludge is fed continuously into a rotating kiln and heated to about 280° C in direct contact with coke derived from the acid sludge, the coke having been ignited to red heat (600° to 650° C) in a separate kiln and returned to the acid-sludge kiln. A simplified flow diagram of the Chemico sludge-conversion process is shown in Fig. 5. The dimensions of the rotary kiln are approximately 80 feet long and 10 feet dia for the heating kiln and 25 × 15 feet for the decomposing vessel. It is claimed that fairly heavy sludges with a sulphuric acid content down to 50 per cent wt can be satisfactorily processed in this system.

The decomposition of heavy sludges containing 25 to 35 per cent wt acid as produced in lubricating-oil treating causes an excessive coke formation, and they are difficult to process. The operation of the decomposing kiln seems to be very sensitive to the composition of the acid sludge, necessitating the composition of the feed being kept as constant as possible. The transport, storage, and injection of the sludge may still present an awkward problem, particularly in the case of a sludge which rapidly solidifies or separates.

The process [7-11] was originally developed by Hechenbleikner, starting from the principle of decomposing acid sludge with hot solids, such as iron balls or sand, continuously circulating between the decomposing vessel and a separate heating furnace. It turned out that the coke produced during

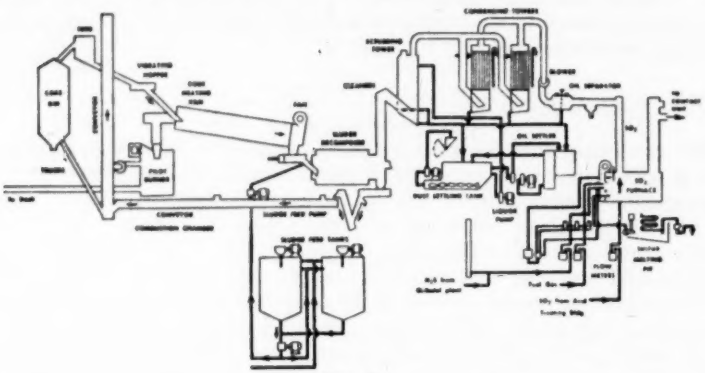

FIG. 5

"CHEMICO" ACID-SLUDGE-DECOMPOSING PLANT

decomposition itself was in a form utilizable as a satisfactory heat-transporting solid.[9] In 1938 a Chemico sludge-decomposing plant started operation at the Philadelphia refinery of the Atlantic Refining Co. The capacity of the plant was 65 short tons of 100 per cent acid per 24-hour day,

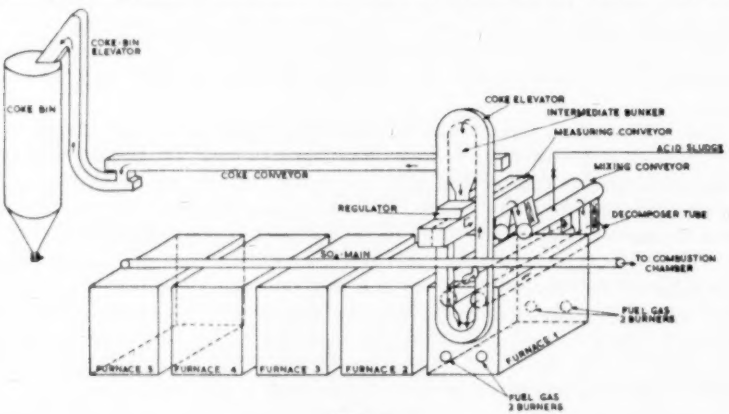

FIG. 6

MONSANTO–ROSS DECOMPOSER PLANT

50 tons from sludge and 15 tons from hydrogen sulphide. A description of the plant was given by Hart.[11]

The *Monsanto–Ross* decomposing system as designed by Monsanto Chemical Co, St Louis, Missouri, U.S.A., is shown schematically in Fig. 6. A unit for the production of approximately 150 tons of sulphuric acid per

day from spent alkylation acid and hydrogen sulphide has been in operation since 1947 at Shell Curaçao refinery.

In this system finely granulated coke, for which initially Dubbs coke may be employed, is circulated continuously through a coke elevator between mixing conveyor tubes and decomposing tubes as shown in the sketch. The mixing conveyors consist of horizontal mild-steel tubes provided with paddles mounted on a shaft. The acid sludge is sprayed on to the coke at the upper end of these conveyors, and is intimately mixed

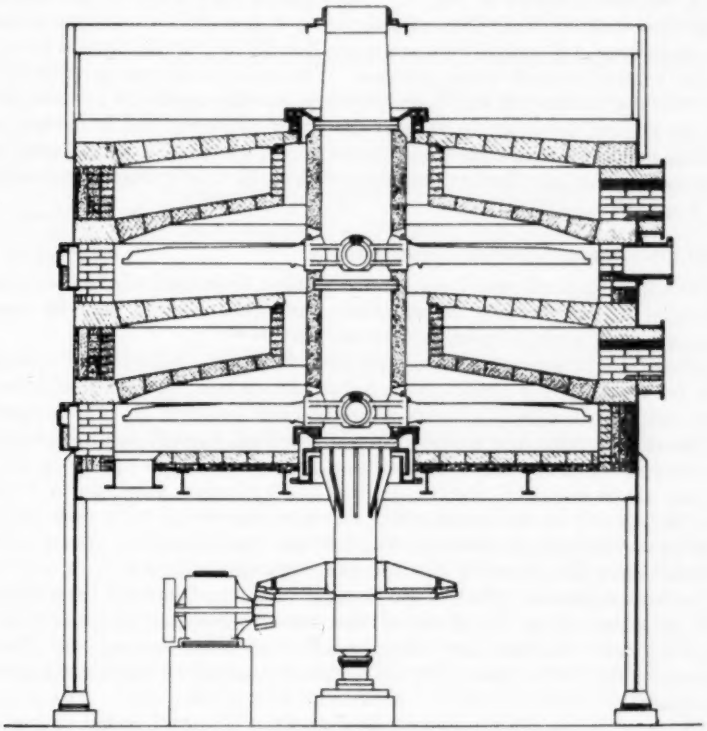

FIG. 7

MONSANTO–MANNHEIM FURNACE FOR ACID SLUDGE DECOMPOSING

with the coke. At the end of the mixing conveyors the sludge–coke mixture drops into the rear end of the decomposer tubes, about 24 inches in I.D. and 20 feet long and provided with conveying paddles. The decomposer tubes are exposed in a gas-heated furnace. The coke temperature inside the decomposer tubes is approx 230° C, the outside temperature of the tubes being about 450° C. The liberated gases are collected in the SO_2 main and are fed to a combustion chamber in order to burn the gaseous hydrocarbons. At the front end of the decomposer tubes the coke drops into a trough and is picked up by the coke elevator, which discharges into the intermediate bunker. The excess of coke, formed during the decom-

position process, overflows on to the main conveyor and is finally dropped into the main coke bin.

The Monsanto unit at Curaçao was installed for the sole purpose of decomposing spent alkylation acid. No more than 15 per cent wt of cracked gasoline acid sludge could be added to the feed.

In the *Monsanto–Mannheim* system a conventional Mannheim furnace is substituted for the decomposing tubes. Less mechanical troubles are expected with this type of decomposing device. A cross-section of a Mannheim furnace is shown in Fig. 7. The furnace comprises two decomposing chambers heated from the outside by hot flue gas circulating between the chambers. A central rotating shaft bearing conveying arms is mounted in the vertical axis of the apparatus. The coke–sludge mixture enters the top chamber at the left and is moved slowly to the centre by rotating arms. At the centre the coke drops into the lower chamber and is moved by a second set of rotating arms towards the periphery of the lower decomposing chamber. The coke leaves the furnace through a hole and is recirculated by a conveying system.

High-temperature Thermal Decomposition

Chemico also developed a conversion method to be used where the primary objective is the recovery of sulphuric acid from sludges poor in organic contaminants, such as spent alkylation acid.[12, 13]

Briefly the process consists of spraying the spent acid with a "spinning-cup burner" into a firebrick-lined furnace, where it is decomposed to form sulphur dioxide at a temperature of 1100° to 1200° C. Hydrocarbons in the sludge, with the addition of gaseous fuel, furnish the heat required to decompose the acid. Where it is desired to make up the acid lost in the alkylation and regeneration cycle, hydrogen sulphide or sulphur can be burned in the combustion furnace instead of carbonaceous fuel. Hot gases leaving the decomposing furnace pass through a silicon carbide tubular heat exchanger of the recuperator type, where heat is recovered by preheating the air entering the furnace. Air, together with the additional fuel, is admitted at the front of the furnace, concentrically around the acid burner. Burners are also installed radially around the furnace perpendicular to the spray of spent acid so as to create a turbulent motion of the gases.

The acid is completely decomposed to form SO_2 and water vapour, the organic material being burnt to carbon dioxide and water vapour. It follows that the yield of SO_2 and finally of recovered sulphuric acid will be higher than in the low-temperature conversion plants, where sulphur gets lost in the coke which is produced as a by-product. It should be noted, however, that the complete combustion process is economically unattractive for converting highly carbonaceous sludges in view of the considerable dilution of the produced SO_2 by carbon dioxide and nitrogen, entailing unnecessarily large equipment in the contact plant.

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ACID SLUDGE—ITS UTILIZATION AND DISPOSAL AT MANCHESTER OIL REFINERY

By R. C. Cobley (Associate Fellow), H. Kaye (Associate Fellow), and R. K. Spencer

The treatment of petroleum products with sulphuric acid to improve their colour, odour, and stability is one of the fundamental processes in refining, and the adoption of newer methods has not seriously affected the total consumption of sulphuric acid by the oil industry. Only a very small part of the sulphuric anhydride consumed in the treating operation finds its way to the market as part of the petroleum product being made, the vast majority of it reacting with a portion of the oil being treated to form acid sludge. This point is well illustrated in Table I, which shows the SO_3 balance at Manchester Oil Refinery.

Table I

SO_3 *Balance at Manchester Oil Refinery*

	SO_3, %		SO_3, %
Intake :		Output :	
As acid	11·4	As sludge	88·3
As oleum	88·6	As sulphonic acids	0·5
		As mahogany sulphonates	0·9
	100·0	As green sulphonates	0·1
		As weak acid	0·6
		Loss	9·6
			100·0

The fact that spent acids from the treatment of such different products as cracked gasoline, petrolatum, and heavy lubricating oils are all known as acid sludge and are identified only by prefixing them with the name of the product concerned, *e.g.*, kerosine sludge, white oil sludge, or lubricating oil sludge indicates the lack of information that exists about their exact chemical composition. The general composition is well known, however, and for the purpose of regeneration, destruction, or disposal, sludge can be considered as made up of five components. These are sulphur trioxide, sulphur dioxide, sulphonic acids, water, and "neutral oils." Still greater simplification reduces this classification to three: mineral acid, organic acid, and neutral oil.

The mineral acid content of sludges can be as low as 35 per cent (calculated as H_2SO_4) when reactive products such as heavy lubricating oils are being treated with small amounts (3 to 5 per cent wt or say 10 to 15 lb per barrel) of low strength, 66° Be and weaker acid. On the other hand, it may rise to over 80 per cent in the later stages of treating for highly refined products such as liquid paraffin, when the acid is used in larger quantities, up to 30 lb per barrel, and in greater strength, as 20 per cent oleum.

The viscosity of the sludge will, in general, be greater, the lower the content of mineral acid, the "thinnest" sludges, and those of highest acid content, being those from gasoline treatment, alkylation processes, and the last stages of liquid paraffin and white oil treatment.

The sulphonic acid content varies considerably, it being greatest in those sludges resulting from processes using high-strength acids, as for example, the production of liquid paraffins and white oils. These sulphonic acids are preferentially water-soluble and are known as green acids to distinguish them, very roughly it may be remarked, from the oil-soluble mahogany sulphonic acids. In quantity they vary from 1 to 30 per cent by weight of the sludge.

The water content results from the liberation of water in the sulphonation reaction, and is frequently the primary reason for the discard of the acid sludge while it still contains large amounts of sulphuric acid, the strength having fallen, due to the liberation of water, below the lower reactive limit. Water (in excess of that combined in the H_2SO_4) may vary between 5 and 20 per cent by weight.

The "neutral oil" so called is composed of those hydrocarbons, and hydrocarbon derivatives which are not describable as sulphonic acids. It includes unreacted hydrocarbons in solution, heavy tar-like polymers and highly condensed hydrocarbons, sulphones and sulphates. Its chemical composition is diverse and largely unknown, and varies enormously, dependent on whether the parent material is a light material such as gasoline, or a heavy fraction of petroleum such as lubricating oil, or even a long residuum.

Typical analyses of sludges obtained during the manufacture of a white oil when the oil was subjected to five successive treatments with 10 per cent by weight of 20 per cent oleum in each treatment are shown in Table II.

Disposal of the large quantities of sludge produced by the average lubricating oil producing refinery can be effected by a variety of methods, dependent on the size of the problem and the resources of the refinery. The simplest is to dump the sludge in open pits and allow wind and weather

to change it from a highly acid, highly viscous liquid to a solid pitch which can be dug out comparatively easily and burnt with other solid fuels under boilers. This method is obviously impracticable where the quantities to be disposed of are very large and the sludges are high in acid and low in hydrocarbon content. In such cases, *e.g.*, alkylation spent acid, or gasoline acid sludge, thermal decomposition of the sludge to form sulphur dioxide and the subsequent production of sulphuric acid by the contact process is a possible answer. However, the cost of these plants in both first cost and operation is high, and they do not normally offer a solution unless the annual consumption of sulphuric acid is in excess of 15,000 tons per annum or the cost of acid is, perhaps due to the refinery's geographical position, abnormally high. To-day one other factor, namely scarcity of acid must also be taken into account.

TABLE II

Analyses of White Oil Sludges

Treatment no.	1	2	3	4	5
	% by wt				
Neutral oil	15	9	9	2	3
Water	9	10	9	8	8
Sulphuric acid . . .	46	49	55	66	77
Sulphonic acid . . .	30	34	27	24	13

Disposal by neutralization and dilution with water is not often practicable, the operation being expensive in chemicals and unsuited to viscous sludges, and the complete removal of the neutral oil fraction proves very difficult owing to the ease with which stable emulsions are formed through the presence of sulphonates. Destruction by burning as a liquid fuel is yet another method, the sludge being burnt alone or diluted with fuel oil.

At the Barton refinery of Manchester Oil Refinery Ltd., the size of the problem is about 12,000 tons per annum of mixed acid sludge resulting from the treatment of lubricating oils with 98 per cent acid, and technical white oil and liquid paraffin with oleum. The major portion of this sludge is disposed of by burning under a range of steam boilers, the balance being used for by-product manufacture.

FUEL FOR STEAM GENERATION

The quantity of sludge to be disposed of is such that dilution with oil was considered undesirable, since it would call for increased capital outlay and result in steam production in excess of the refinery needs. The sludge burnt has an acid content of 40 to 75 per cent by weight and a calorific value of 4000 to 6000 B.Th.U. per pound. It is normally a free-flowing liquid, but occasionally may be of high viscosity.

Sludges from the various treatments fall into a common receiver, whence steam-driven reciprocating pumps move the mixture to the oil-segregation section of the disposal plant, where any oil sent over can be drawn off. This section consists of four unlined vertical mild-steel tanks each with a capacity of 12,000 gallons. Three tanks are used alternatively for filling, settling (for oil separation), and burning, while the fourth tank is used to

accumulate batches of recovered oil. At present this system is worked on a comfortable 24-hour cycle. At the end of each 24 hours the tank which has been settling is put into service as the burner supply tank (during the previous 24 hours this tank will have been "settled," its oil drawn off, and the remaining sludge will have been vigorously circulated for at least 2 hours). The tank which was the burner-supply tank is switched to receiving, while the third tank, which was previously on receiving, is now switched to settling. Provision has been made for pumping back from the

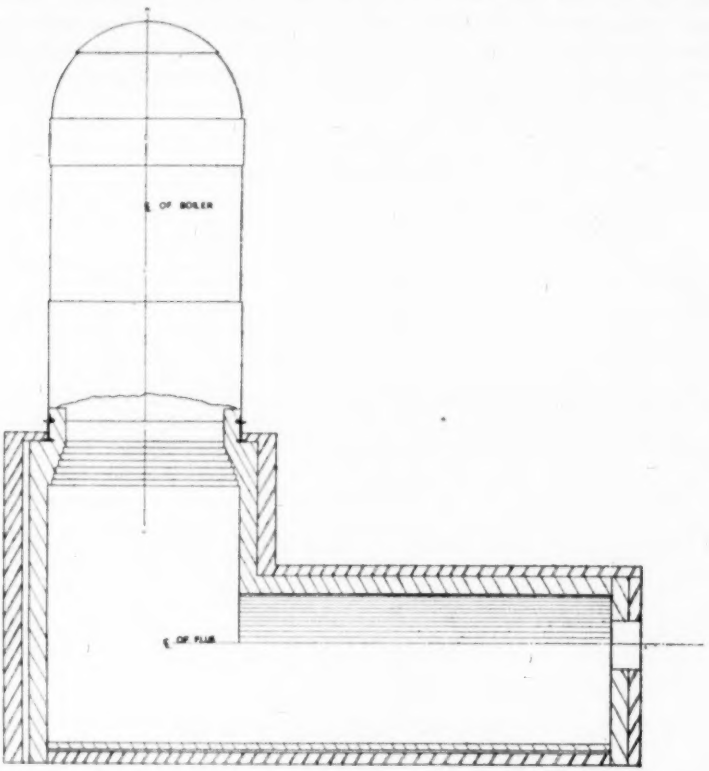

FIG. 1

RECTANGULAR FURNACE

recovered-oil tank any sludge which may have formed in the tank, or which may have been inadvertently pumped into it when drawing off recovered oil.

Oil is recovered from the top of the settling tank by means of a swing arm; test points and an experimental interface level indicator being provided for the guidance of the operator. To avoid tedium of operation the pumping system has been manifolded so that all operations take place at ground level. No heating is provided in tanks or on pipelines. All tank openings are weatherproof.

Special attention has been given to ensuring that the tank on burner supply can be adequately mixed. The more viscous sludges reduce burning rate, while certain of the lighter sludges cause carbon particles to be broadcast from the furnace stack. These particles of carbon, being large in relation to weight, can be carried quite long distances, and would be the cause of justifiable complaint. It has been established that good atomization alone will not eliminate carbon particles, the answer to this problem lies in thoroughly mixing the sludge prior to burning. Mixing of the tank is achieved by circulation through a system independent of the burner supply system. In addition, some circulation is achieved by the burner supply system in which the sludge is pumped from the base of the tank through a ring main, past the burners, back to the top of the tank.

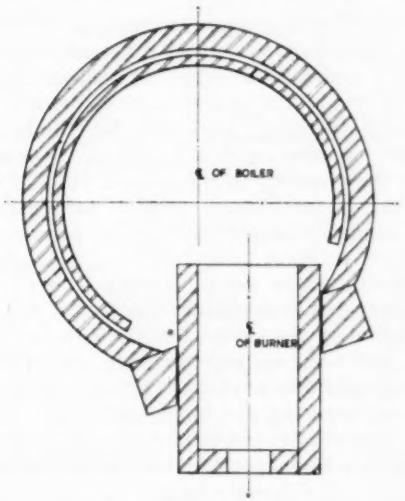

FIG. 2
CIRCULAR FURNACE

The ring main ensures that the head of the sludge available at the burners is a constant 40 feet.

The pipelines handling sludge are generously proportioned, as sometimes the sludges available are heavy and a flow is maintained only with difficulty. One feature of the sludge pump worthy of mention is the type of valve in use at the plunger end. This is a heavy ball type valve of cast iron, which since its adoption has improved pump performance and greatly reduced maintenance costs.

A large furnace volume is necessary to complete the burning of acid sludge. Originally the burning arrangement consisted of a rectangular fire-brick structure in the form of a dutch oven with a bonded, arched roof, Fig. 1. Experience had enabled the furnace to be proportioned to give the necessary heat to burn the maximum amount of sludge without too much stress on the brickwork. The proportions of the furnace were 0·4 length = breadth = height, with 1·4 cu. ft. of furnace volume provided per pound

of sludge burned per hour. This large furnace-volume allowance was necessary for two reasons: (1) ample space for excess air under natural draught conditions, and (2) the low heat liberation rate (about 4000 B.Th.U. per cubic foot furnace volume) requires a hot combustion chamber without undue overheating of the brickwork at the maximum rate of firing. The walls and arch were made purposely as thin as safety would permit, to help in cooling the brickwork.

On top of the furnace at its end remote from the burner, a vertical-type fire tube boiler was mounted on steel framework. This was a compact arrangement whereby firing and water tendency were carried out by the operators at one level. The boiler may be regarded as an ordinary type used for utilizing waste heat, but some advantage was gained from radiant heat.

Although furnaces of the dutch-oven type are still in operation, the latest furnaces have been built to a rather different pattern in an endeavour to improve burning rates and reduce structural faults. These later furnaces more closely approximate to an up-draught type. The first consisted of a vertical cylindrical brick chamber, the boiler being mounted vertically above the chamber on steel framework at a height sufficient to corbel easily from the chamber to the circular skirt of the boiler, Fig. 2. The circular section of the furnace permits tangential firing, increases turbulence, and generally improves burning rate, but the structural improvement was not so great as had been anticipated. It was thought that since the structure was self-supporting it would not be seriously affected by movement due to expansion: in fact, it was, and the difficulty was overcome by building the second furnace with an octagonal instead of a circular section, and a flat suspended roof in place of the corbelled cone, Fig. 3. The octagonal section permits allowances for expansion to be made, whilst still maintaining the tangential firing. Firing is through a short tunnel set tangentially to the furnace, the walls of which are protected from impingement by a thin screen wall which can be quickly and cheaply repaired as required. The furnace volume provided per pound of sludge burned per hour has been reduced to 0·7 cu. ft.; height and diameter of the furnace being approximately equal.

One burner is used per furnace. Its fittings are made up from ordinary pipe material and the burner consists of an outer tube for the passage of sludge with a central tube for steam. The small-bore central tube is set back from the open end of the outer tube, and the steam from it injects the sludge into the furnace in the form of a round, slightly divergent spray. This type of spray has been adopted in an attempt to keep as much sludge as possible from striking the side walls and floor before burning is completed. It has been found impossible to avoid this altogether, and burning on these areas causes some wasting of the brickwork.

The steam used for atomization of sludge is about 0·35 lb per lb. This represents about 15 per cent of the steam generated by the boiler, and, although relatively high, is compensated for by the fact that the steam injected into the furnace plays its part in aiding complete and smokeless combustion.

A burner using fuel oil is fitted to the furnace, and is employed when starting up. It is sometimes necessary to use fuel oil when the sludge is very heavy

and cannot be injected into the furnace in sufficient quantity to maintain temperature. Normally, with well-mixed sludges, the fuel oil is not needed.

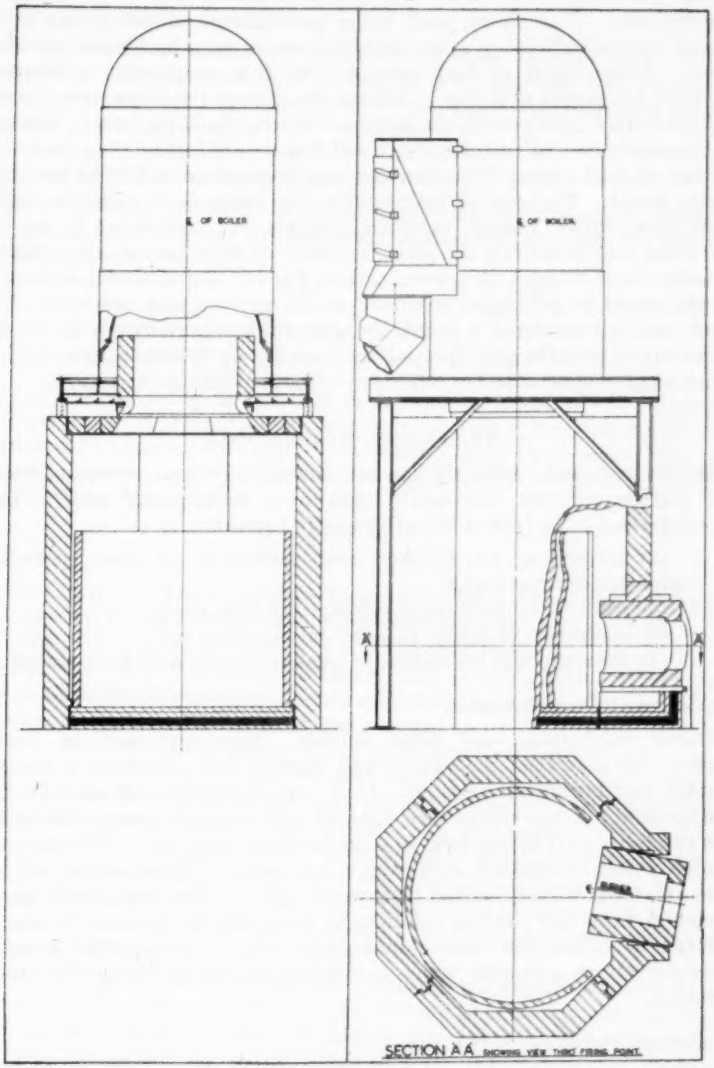

FIG. 3

OCTAGONAL FURNACE

The furnace temperature for best burning conditions is about 2100° F. This temperature is easily reached when burning the maximum quantity of well-mixed sludge. About 80 per cent excess air is required to keep the

brickwork from overheating at this burning rate. Overheating will occur at reduced loads if the percentage of oil entrained in the sludge rises.

Exit-gas temperature from the boilers is about 600° F at maximum burning rate. This shows good boiler performance, although the actual overall thermal efficiency is not high because of large radiation and stack losses. About 2·5 lb of feed water at 100° F is evaporated to steam at 100 p.s.i. per pound of sludge. Taking the average heating value of sludge as 5000 B.Th.U. per pound, the thermal efficiency is 55 per cent. As might be expected, cost of maintaining these furnaces is higher than that for a normal oil-fired setting of similar size, and averages around 2*s* 6*d* per ton of sludge burnt. The cost of maintaining the whole unit, including boiler, oven, flues, tanks, pumps, pipelines, burners, etc., is around 7*s* per ton. The total cost of running the plant, inclusive of depreciation, amortization, or other fixed charges, is approximately 29*s* per ton of sludge consumed. As the steam so generated is valued at 31*s* per ton, the operation of the plant can be considered a profitable undertaking, particularly if the only alternatives possible are disposal by burying or neutralization, both of which require substantial expenditure without yielding any return.

By-product Manufacture

Whilst acid and oleum sludges are mainly used as a source of fuel at the Barton refinery alternative uses have been investigated. These investigations have been directed generally towards :—

(*a*) extraction, purification, and utilization of green petroleum sulphonates from sludge;
(*b*) recovery of mahogany sulphonates from sludge;
(*c*) utilization of sludge in white oil manufacture;
(*d*) recovery and utilization of weak sulphuric acid from sludge.

Green Petroleum Sulphonates

Green sulphonates are water soluble; they are used as wetting agents and industrial detergents, and have a high efficiency in lowering surface and interfacial tensions. Only certain sludges are used for their preparation. These sludges are digested with water to remove the bulk of the sulphuric acid in the form of a 35 to 45 per cent acid. The extracted sludge is then neutralized with caustic soda and the green sodium sulphonates extracted with industrial methylated spirit. The methylated spirit is removed from the extract solution by evaporation, leaving an aqueous solution of sodium petroleum sulphonate which can be marketed as such or alternatively as a powder which is obtained by spray drying the aqueous solution.

Mahagony Petroleum Sulphonates

Mahogany sulphonates are normally to be found in the oil which has been subjected to oleum treatment, but some sulphonates which may yet be classed as mahogany are present in the sludge. These mahogany sulphonates can be recovered by extracting the sludge with various petroleum fractions which act as solvents for the various sulphouates. The properties of the sulphonates thus obtained are dependent upon the

solvent used, but in general they are good-quality mahogany sulphonates and have high emulsifying power.

Sludges in White Oil Manufacture

Sludges obtained in the later stages of white oil manufacture, *i.e.*, those containing high percentages of sulphuric acid, may be used in place of oleum to carry out the initial treatments, thus constituting a form of counter-current process.

Extraction and Utilization of Weak Acid

The weak acid produced by the aqueous extraction of oleum sludges in the manufacture of green sulphonates is dark in colour or has the following typical analysis :—

	% by weight
H_2SO_4	40
Sulphonic acid	2
Ash $\{Na_2SO_4, Fe_2O_3\}$ . . .	0·15

Such an acid could be used for the treatment of distillates in place of the conventional strength acid, but only if high temperatures are used. Materials of construction limit development along these lines. The acid is used, however, as a " pickling " acid, and a further use may be found for it in fertilizer manufacture.

Discussion

The Chairman, introducing the discussion, said : A century ago kerosine was distilled from petroleum and marketed for illuminating purposes. The chemical that was used to refine it was sulphuric acid.

Later the internal-combustion engine was developed, and with it arose the requirement for gasoline. Again sulphuric acid was used as the refining agent as and where such refining was necessary.

So, as the number of products has increased from year to year, the oil refiner has turned first of all to sulphuric acid for his treatment operations.

Oil refiners have constantly sought technical improvements, and various other treatment methods have been brought into practice which do not involve the use of sulphuric acid. In the case of the light products, such as gasoline, white spirit, and kerosine, the sulphuric acid method has been largely superseded by other methods. In the case of lubricating oils other treatment methods have been evolved, but these have not superseded completely the sulphuric acid method.

Lubricating-oil solvent-extraction methods are mainly concerned with viscosity-index improvement and improvement in oxidation characteristics. Such results cannot be obtained always by the sulphuric acid method. On the other hand, oils in this range such as technical whites, medicinal paraffin, and highly refined electrical oils, can only be refined by the sulphuric acid method. Solvent-extraction methods are not sufficiently drastic. Again, solvent extraction is a more expensive process than sulphuric acid treatment. Where high viscosity index and a good oxidation test are not specific requirements, the latter method is generally utilized.

Solvent-extraction methods have provided the oil refiner with an

additional tool which has made it possible for him to produce high viscosity index, good oxidation test lubricating oils. As such they have opened up new refining possibilities rather than superseded requirements formerly covered by sulphuric acid treatment.

It is thus apparent that in the straight refining of petroleum products, the main requirement for sulphuric acid treatment is in the lubricating-oil range and mainly for highly refined products. It is also evident that unless further new methods are evolved this state of affairs is likely to persist.

The sulphuric acid treatment of lubricating-oil distillates for highly refined oils results in a high acid usage and a high resultant make of acid sludge. This acid sludge is a complex mixture of acid and hydrocarbons varying from liquid sludges to sludges which rapidly solidify, and from high acid content to high hydrocarbon content. These particular sludges have always provided a problem to the oil refiner.

Large-scale refining in so far as the United Kingdom was concerned was, until recent years, largely carried out abroad. There the refiner could, to a large extent, follow the designer's instructions, which consisted mainly of an arrow pointing downwards from the base of the agitator. Acid was a comparatively cheap commodity, and the economy of its recovery was not apparent. With the rise of large-scale refining in the United Kingdom, the refiner had to revise his disposal ideas.

Acid manufacturers were not greatly interested in the oil refiner's problem. They could probably produce acid cheaper from raw sulphur than from the oil refiner's waste material or, alternatively, they had not the requisite plant to handle this material.

On the oil refiner's side though his acid sludge make was comparatively high, it was in no case sufficiently high individually to meet the minimum yearly tonnage requirement to justify the erection of an acid-recovery plant.

Development in the United Kingdom has thus followed the line of utilizing the fuel value of the acid sludge. This has either been carried out under the main boilers, to the disgust of the engineers concerned, or in specially devised furnaces.

The expansion of the petroleum-refining facilities in the United Kingdom includes the erection of alkylation units. This is a chemical process utilizing sulphuric acid. It results in a waste acid with low hydrocarbon content. In this case the recovery of the acid is a simpler problem, and one which is more or less necessitated by the acid usage. It is probable that refiners with alkylation on their refinery expansion programme will cover the question of acid recovery to include all acid sludges.

During the last year a shortage of raw sulphur has developed. This has made it desirable for the oil refiner to review his usage of sulphuric acid and the potentialities of its recovery from his waste products. Individually, except where alkylation units are projected, the recovery of the waste acid offers a difficult problem to the home refiner. To develop his potentialities, co-operation of some kind is necessary.

This symposium has been organized so that present disposal methods can be reviewed, and the possibilities of lowering the oil refiner's sulphur requirements can be discussed.

C. I. KELLY: The authors of the four papers in this symposium deal with a refinery by-product which has always caused refinery operators considerable trouble.

The subject of acid-sludge disposal and utilization is one which also touches sulphur closely. The world shortage of sulphur to-day is critical, as it is the main source-material for the manufacture of sulphuric acid. It is therefore a matter of national interest that wherever possible the sulphuric acid content of waste sludges be returned to industry for re-use, contributing in that way to an easement of the general critical world situation.

Obviously, the short-cut road to having no disposal problem is not to use sulphuric acid.

A cursory glance at the U.S. petroleum industry shows that the non-use of acid by it is an impossibility; it also indicates that in the course of the past twenty years two things have happened in the U.S. in this matter:

(1) The rate of acid consumption, expressed in terms of 100 per cent acid used per unit amount of crude charged to the stills has fallen.

(2) The overall weight of acid used has remained substantially uniform at about 600,000 tons per year, or about one-third of the U.K. annual production.

However, in line with the U.S. tendency in recent years to stress to the public how it serves the community, it has recently been made known that the U.S. petroleum industry is a net producer of acid or equivalent sulphur. During 1951 the U.S. petroleum industry will consume about 638,000 tons of sulphur and at the same time release about 643,000 tons of sulphur to industry outside itself; that is, it will be a net producer to the extent of about 5500 tons—a figure shortly to be increased, it is said, to about 190,000 tons.

The petroleum industry has been able to do this by improving the way in which acid and raw product are contacted; by evolving a scheme under which incoming acid is degraded in successive stages into an almost impossible sort of sludge; by replacing acid processes with those of solvent extraction; by utilizing its hydrogen sulphide by-product for the making of acid or conversion into sulphur; and by treating waste acid and acid sludge in order to return good acid into its own refinery operations.

In the U.K., on the other hand, the petroleum industry consumes about 60,000 tons of sulphuric acid per annum, or 20,000 tons of sulphur equivalent per annum.

In return, by the utilization of the hydrogen sulphide from petroleum operations, the U.K. petroleum industry will soon be handing over to the U.K. chemical industry at least 25,000 tons of sulphur per annum.

There remains the question, what can the U.K. petroleum industry contribute in addition towards an amelioration of the position of the nation's sulphur and other acid-making raw materials?

That question will largely be answered by the outcome of the discussion on this occasion, and by steps that may be taken eventually not to burn or in other ways wastefully dispose of the acid or sulphur content of the acid sludges in U.K. refineries.

A way which may not altogether be acceptable, because it is perhaps divorced from essential practical considerations, is suggested by some of the authors :

(*a*) Squire, *et al*, in stressing the need for a method suitable for the handling of about 20 tons per day of diverse tars, as a means to enable the small refiner to recover acid, say : " Unless recovery of the acid tar output from a number of refineries could be arranged, at present there seems little real alternative to disposal by destruction."

(*b*) Mr Cluer, in the last paragraph of his paper, brings up the same point in reference to acid sludge at Ellesmere Port, and he says : " The most constructive approach to this problem appears to be joint operation of, or supply of sludge to, a sulphuric acid plant by two or more suitably located refineries." This he says because his 7000 tons of sludge, containing 1300 tons of sulphur (combined), is too small a quantity to be handled alone.

(*c*) Cobley, *et al*, touch on the same theme. They say the cost of a sludge-decomposition plant leading to sulphuric acid manufacture is too high in both first cost and cost of operation, " and they do not normally offer a solution unless the annual consumption of sulphuric acid is in excess of 15,000 tons per annum of acid. . . ."

My concluding remarks take the form of a suggestion that perhaps some such co-operative venture will be possible in one or two, perhaps three, regions in the U.K. when the whole refinery expansion scheme is completed, in each of which regions two or three sources of acid sludge may be found to justify *in toto* joint operation for the profitable disposal of acid sludge. For example, according to rough estimates, I find that in the northwest region between Ellesmere and Trafford Park the amount of acid sludge at present made may be between 25,000 and 36,000 tons per year, containing from 15,000 to 18,000 tons of acid expressed as 100 per cent acid. This is equivalent to about 5000 to 6000 tons of sulphur per year or, in other terms, 50 to 60 tons of acid sludge with a potential of 16 to 20 tons of sulphur per day.

The arising question is, can such regional sources of acid sludges of various kinds be combined and worked up into usable acid and in this way again contribute to an easement in the position regarding sulphur and sulphur-making raw materials ?

Dr J. S. Carter (Deputy Chief Alkali Inspector, Ministry of Local Government and Planning), after expressing the regret of the Chief Inspector, Mr W. A. Damon, that he was unable to be present, said : The national sulphuric acid supply position is now such that I feel the petroleum industry should do all in its power to recover acid from sludge, even though the operation is undoubtedly difficult and perhaps unremunerative. Burning of sludge, even when disguised as " thermal recovery," is to be deplored. I do not underestimate the difficulties. The operation of a contact sulphuric acid plant using sludge as the source of sulphur is not easy, and methods for the recovery of dilute acid yield a product looked upon with disfavour by potential users. Moreover—and especially when the original sludge contains sludges from refining of coal-tar products—

the recovered acid contains odorous impurities, so that when used in any operation involving evolution of gases, *e.g.*, hydrogen when used for pickling of steel and carbon dioxide when used for dissolving of carbonates to produce sulphates, the emission might be so offensive as to require treatment if local complaint is to be avoided. Nevertheless, I feel the industry should persevere. I suggest an approach to the sulphuric acid industry and mention the new anhydrite sulphuric acid units to be erected where the two products are acid and cement clinker and where there is to be close collaboration between acid and cement interests.

If recourse must be had to burning, this should at least be done properly. There should be complete combustion, freedom from escapes at low level, and the final discharge should be at as high a level as practicable. An important factor is the mass rate of emission of sulphur dioxide. Assuming that 30 tons of sulphur dioxide could escape daily from a large power-station from a chimney 300 ft in height without serious contamination of the air at ground level some ten chimney lengths distant and with a normal breeze, the proportionable allowable rates and chimney heights would be

300 ft	30 tons SO_2 per day
250 ft	21 „ „
200 ft	13 „ „
150 ft	7·5 „ „
100 ft	3·0 „ „

In considering the above, the assumption is made that the whole of the sulphur dioxide is discharged from one point source. Each case must, of course, be considered on its merits and due allowance paid to prevailing winds, contours etc., but the above is my Department's "yardstick." This yardstick does not replace the older ruling relative to concentrations, and indeed the two are to some extent complementary, especially for lower chimney heights. The aim should still be that at 100 ft the total acidity of the escaping gases should not exceed the equivalent of 1·0 grain calculated as sulphur trioxide per cubic foot. The foregoing relates to hot dry gases free from mist. Cold or wet gases would need special consideration. The total acidities recorded by my Department for chimneys taking gases from burning of sludges had ranged up to 20 or more grains per cubic foot in the above units, with more recent figures showing a variation of from 1 to 5 grains per cubic foot. The average acidity of all escapes from all processes registered under the Alkali Act over the past twenty years or so is about 1·3 grain per cubic foot.

R. Robinson: We have endeavoured to make a contribution towards acid recovery from acid sludges, but so far we are meeting with the difficulty of finding sufficient assured supplies of acid sludges of the right types for use in the Monsanto–Ross–Wilde process.

What is the economical capacity of plant? From the information we have it seems that the smallest unit at present used is one with a capacity of 25 tons acid per day from sludges of the right type. The capacity of the plant will vary with the water content and oil content of the sludge, and the same plant may run at as low as 15 tons acid per day if the oil content of the sludge is high.

DR H. E. NEWELL: What is the concentration of SO_2 in the exhaust gases from furnaces burning acid sludge?

DR CARTER: The concentration has at times exceeded the equivalent of 20 grains or more calculated as sulphur trioxide per cubic foot—which I hope is abnormal—but it can be reduced to somewhere between 3 and 4 grains. The average of the emissions from all chimneys in the U.K. which have been tested over the past twenty years by the Alkali Department is about 1·5 grains per cubic foot. The SO_2 concentrations from burning of acid sludge are not normally such as make SO_2 recovery an attractive proposition.

F. E. WARNER (Carless, Capel & Leonard Ltd.): We have had an acid problem for the last ninety years, and it does not get any better; indeed, it gets worse as time passes. We have coal-tar material as well as petroleum to treat. Part is processed in batch plant, and for the last twenty years we have had a continuous washing plant of the centrifuge type as well. The acid sludge amounts to about 1000 tons a year, and our difficulties are probably paralleled by many others in the coal-tar industry.

We are interested to hear that co-operative schemes for disposal are being considered. We have always tried to carry out our own disposal without burning the sludge, and have received much assistance from the Alkali Inspector. Every process has its difficulties, partly because of the scale of operation; the quantity is large enough to be a considerable nuisance, but too small for any satisfactory recovery process.

Some of the methods used have been mentioned by Dr Carter. We tried to find an outlet through superphosphate manufacture, but the product is too smelly to be acceptable. We made ferrous sulphate for sewage treatment, but this is no longer required. The people who pickle steel tried to take the acid, but gave it up. For some time we have operated a subsidiary works for secondary metal recovery, in which the acid was used for metal beneficiation, raising the copper content of various brass residues. The change in the market price of zinc no longer allows the purchase of material on its copper value alone, and the removal of zinc is not easy in any case. The hydrogen evolved reduces sulphur compounds and gives a smelly gas which is difficult to burn safely when air is drawn in with it. The zinc sulphate produced cannot be purified owing to contamination with material from the coal-tar products. We changed over to neutralization with lime, but the disposal of the sludge in the London area becomes increasingly difficult.

Some reduction in the amount of sludge to be treated has come from the re-use of the acid in successively degraded stages. We have also cleaned up sludge by dilution and used the resulting weak acid for base extraction. The tar has gone to provide fuel for melting up recovered metals.

The logical method of sludge treatments is to recover acid, either by reconcentration of acid which has been diluted and cleaned up or by complete decomposition to SO_2 and reconversion. The smallest acid reconcentration plant is a pot-still unit which can run on 10 tons per day. Complete decomposition with catalytic re-conversion involves wet and dry

electrostatic treaters and a complicated plant. It might be worth considering conversion of gases from decomposition in a chamber plant.

All these methods, however, are on a scale for which our production is too small.

DR F. SAGER: In several European refineries acid sludge from the acid treatment of lubricating oils is hydrolysed in the presence of tar oils in order to facilitate its solution and the separation of the oil and aqueous phase.

I have also seen the case where a light solvent was added during hydrolysis. This was later recovered by distillation. The products of hydrolysis were a diluted sulphuric acid of about 40° Bé and a bitumen. Has anybody in this audience had a similar experience?

The amount of water added for hydrolysis of an acid sludge plays an important role. One aims to obtain an acid of as high a concentration as possible in order to use it directly in chemical industry or, where further concentration is required, to do this operation at the lowest possible cost.

General practice has shown that in order to facilitate phase separation a greater percentage of water has to be added to an acid sludge than is strictly required for carrying out hydrolysis. As an example I can give data from the recovery of acid sludge from the acid refining of shale distillate. By dilution with water in plant operation an acid was obtained which did not contain more than 10 to 15 per cent H_2SO_4. By using mechanical means of separation immediately after diluting the sludge it has been possible to reduce the quantity of water to such a percentage as to allow the recovery of an acid containing as much as 50 per cent H_2SO_4.

THE CHAIRMAN: I think we obtained a bitumen resin on a small scale after hydrolysing acid sludge.

MR CLUER: The bitumen-like residue referred to in the paper was produced after driving off SO_2 alone, without the need for prior hydrolysis.

THE CHAIRMAN: We produced an excellent resin from a bitumen. But all I can say is that the penetration was zero, and we are up against the problem of what to do with it.

T. H. STOTHARD: With reference to the paper by Mr Nieuwenhuis, I was interested in the method of producing a white acid from concentrated sludge acid by using nitric acid for the oxidation of organic material. Has this method been used in practice, and, if so, could we have some indication of the economics of the process? For example, do we require a large amount of nitric acid for, say, 100 parts of sulphuric acid?

MR NIEUWENHUIS: The amounts of nitric acid used are small, provided the concentration of the acid is not too low. The remark made in the paper refers to concentrating black acid, where the final product was not up to the standard in respect of colour, but the total amount of carbon present in the acid was very low. Also, filtration was helpful in that case. The addition of nitric acid as a general method is not covered by the remark

in the paper; it refers to the case of concentrating acids containing not too large quantities of carbon. The effect of nitric acid addition on oxidation of organic contaminants should be investigated experimentally for each particular case under consideration.

MR WARNER: The percentage of acid normally added to the hot acid coming out after treatment is about 1 per cent.

C. C. SMITH: It is understandable that the meeting has concentrated rather on the problem of dealing with the acid. But there is another aspect. A problem that also affects us is that of recovering usable oils from the acid tar itself. Is the process, as carried out in the petroleum industry, so efficient that there is never any oil from the original process present in the acid tar? I should like to think that that is so; but if it is not, is not some special treatment involved? That is not referred to by any of the authors of the papers.

Then there is this question of dealing with the acid tar—we have had some experience of separation after dilution, to which the authors of the papers have referred—and whether or not, by the addition of further oils, there is a probability of producing a pitchy material. When no further oils are added, all that is left after separation of the diluted acid is a solid pitchy residue, that can be handled in the way that most products of low penetration are dealt with.

THE CHAIRMAN: Comment has been made about the lack of research on the hydrocarbon content of acid sludge. It is probable that comprehensive research would produce interesting results. Acid sludge has, however, been at all times the Cinderella of the oil-refining business. The tendency has been for the refinery manager to pass the problem to the chief engineer or the transport manager for suitable disposal arrangements. Where the problem has been passed to research, the initial indications of costs and profitabilities have not been attractive enough to justify further research expenditure.

Comment has also been made about hydrolysing. The simplest method I have seen was in operation in a French refinery. The acid treatment of lubricating oils was by the conventional agitator method. The flow was in the vertical with the agitators on the top floor, down through the settlers and the clay-treatment plant to finished products on the ground floor. From the base of the agitators hardwood open-top troughs ran out through the building wall to the top of two vertical tanks. The acid sludge gravitated along these troughs into the tanks. The tanks had a water bottom and live-steam injectors.

The dilute acid was drawn off and returned to the acid manufacturer. The hydrocarbon was dropped down into an open pit with an ashes bottom and allowed to harden.

The hardened sludge was dug out and transported by wheelbarrow down the hill to the boiler-house, where it was stacked. The firemen shovelled the hydrocarbon on to the coal-fired chain-grate stokers, which were lipped at the edge to prevent overflowing into the ash pit.

The refinery had an agreement with the acid manufacturer stipulating

that purchase of fresh acid was based on their acceptance of the waste acid.

The method was crude, but it was effective. Perhaps the French are more realistic about such matters than we are.

DR R. SPERLING: We have found that the more viscous sludges contain a small percentage of entrained oil which is very difficult to remove even on prolonged centrifuging. Some of this oil can be recovered by mixing the viscous sludges with the more fluid highly acidic sludges.

DR A. L. DOWN: In the case of a fairly large refinery, where a sludge-decomposition plant is installed for the manufacture of sulphuric acid, the plant can only operate on sludge that can be pumped to the unit. On standing, the sludge from the acid treating of cracked distillates deposits polymer which cannot be pumped. What methods are used for handling this polymer?

M. GAUL: Our experience in Abadan is confined to acid sludges derived from treating fractions from Persian crude oils ranging from light gasoline to kerosine. Acid treatment is carried out with sulphuric acid (98 per cent wt) or alkylation spent acids (88 to 92 per cent wt H_2SO_4). We have no sludges derived from acid treatment of lubricating oils.

We have never experienced any trouble with heavy deposits in the plant tanks.

The various sludges are collected in tanks sited near the acid washeries in order to separate any oil coming forward with the sludge. Sludge is then pumped to the tanks at the decomposition plant and circulated before and during feeding to the decomposer. The object of circulation is to give homogeneity to the feed and distribute throughout the sludge any oil which has escaped previous separation. Circulation can be intermittent or continuous, continuous is preferred.

DR J. L. EDGAR: The point has been made that, if you have a sludge decomposer you should also have alternative sludge-disposal facilities in order to keep the rest of the plant in operation during periods when the decomposer is shut down for maintenance. Can Mr Gaul give us an idea of the sort of difficulties that are experienced with this type of equipment?

MR GAUL: We have a cold circulation system, and I think that is the biggest difficulty. We have a heating kiln for the material going to the decomposer, and we have an elevator to the storage bins. We have had difficulty with that, and again we have had a lot of difficulty with mud circulation.

MR EDGAR: Do you have much difficulty with the decomposer itself?

MR GAUL: No; normally that runs without much trouble. It is more in the circulation of the materials than in the operation of the kiln that we experience trouble. The coke itself contains very little residual acid and leaves the decomposer quite dry. The atmosphere in the decomposer consists largely of sulphur dioxide, and we rely on a coke seal to prevent

this getting into the coke conveyer. Some always passes forward with the coke itself and, when the plant is shut down, steam can be bled into the conveyor to prevent combustion of the hot coke if air leaks occur. As the plant cools down the weak acid formed by the acid gases and condensation can cause corrosion.

V. BISKE: In connexion with analyses of acid sludges I notice that in the paper by Cobley, Kaye, and Spencer, the water contents quoted for the sludges are considerably higher than those given by Squire, Pidgeon, Jones, or by Cluer. Could such differences perhaps be attributed to the analytical procedure used? It would be useful to have some information concerning the test methods employed in the various cases.

I have another query, which I would address to Dr Carter. He has mentioned the recent Order (No. 364 of 1950) by which acid-sludge works are brought within the provisions of the Alkali Act. As I see it, petroleum refineries have, in this respect, been covered by the Act since at least 1935, as the Order made in that year (No. 162) added to the definition of scheduled works, "works in which any product of the refining of crude shale oil or crude petroleum is treated so as to cause the evolution of gases containing any sulphur compound."

This would seem to be sufficiently wide to include any sludge-treating process when carried out at a refinery, and I therefore presume that the new Order made last year does not alter the position of refineries and is merely intended to cover processing of acid sludge when it is done elsewhere than at a petroleum refinery. Could Dr Carter say whether this interpretation of the regulations is in accordance with the views of his Department?

H. KAYE: The figures quoted in our paper were obtained by a modified Dean and Stark method using zinc oxide for neutralization of the free acid. It is appreciated that this method gives higher values than does the Karl Fischer method. Thus, on a technical white oil sludge (50 to 55 per cent H_2SO_4) the Fischer method gave 4·2 per cent water, the modified Dean and Stark method gave 6·0 per cent water, and on a late liquid paraffin sludge (85 per cent H_2SO_4), 2·3 per cent water was determined by the Fischer method, whereas 5·5 per cent was the value obtained by the modified Dean and Stark technique.

P. C. JONES: At the National Oil Refineries we too use the Dean and Stark aniline sulphate method for determining the water content of an acid tar. But I think that the differences referred to may be accounted for by the fact that the water content is a function partly of the treatment and of the number of stages of treatment, and partly of the percentage of free water present in the feed. Thus the first stage of a countercurrent acid contactor may be regarded primarily as a dehydration stage, while the greater the number of stages, the greater will be the degradation of the sulphuric acid to water and sulphonic compounds.

DR CARTER: As to the question whether the acid-sludge process came under the existing paraffin-oil definition, there was some doubt. The

definition did not include the treatment of acid sludge arising from the treatment of coal-tar products. When we extended the provisions of the Alkali Act in 1950 we included "acid-sludge works," so that we were able to cover the petroleum and coal-tar industries.

D. G. PIDGEON: I would like to put a question to Mr Nieuwenhuis. During the meeting we have heard some experiences of the Chemico type of decomposer at the recovery plant. But the Shell organization use the Monsanto type of plant. Can he parallel for us the remarks made about maintenance difficulties? Our experience with the Chemico plant is that we have had difficulty in maintaining a time efficiency better than 75 per cent.

MR NIEUWENHUIS: Unfortunately the maintenance costs of the Monsanto plant were very high, so that in comparing the Monsanto and the Chemico methods I do not think it is possible to show a preference for either on the basis of maintenance costs. Also in the Monsanto plant the difficulties are mainly caused by mechanical imperfections. I think these difficulties are of a fundamental nature, since we have to handle solid materials under very difficult conditions, and the moving parts will show interruptions sooner or later.

There is, as you know, one essential difference between the two methods. In the Chemico method the acid sludge decomposes in a rotating drum, where the sludge is coked, and the heat is applied in a separate drum where the coke is heated directly by gas. In the Monsanto system the heat is supplied through a metal wall from an outside source, as shown in Fig 6 of my paper, and this was one of the main sources of constant difficulty. In the drawing, two decomposer tubes are shown. Inside the tubes there is a horizontal rotating shaft with paddles which transfers the coke from one side to the other of the tubes as they are heated in the furnace. It turned out that it was essential to heat the tubes evenly from all directions, since, if there were a temperature difference, the tube would bend and the conveyor would jam.

Mechanical difficulties were also encountered in the mixing conveyor located above the decomposer tubes. Here again jamming of the moving parts occurred. Furthermore, severe corrosion was experienced combined with abrasion of the paddles by the coke.

The acid-sludge distribution over the coke also presented a problem. If the sludge were not of the right composition we found it very difficult to mix it with the coke evenly, so that there were lumps of coke of high acid content, which gave rise to severe corrosion and incomplete decomposition in the decomposer tubes. Further, numerous problems were encountered with the conveyor system such as jamming of coke with consequent breaking of shafts or transmissions.

Summarizing, I do not think this method has yet reached the stage of development at which it can be recommended as being the best method; but at least it works on spent alkylation acid. We were never successful when trying the Monsanto method on heavier types of acid sludge. From what has been said to-day on the Chemico rotating-drum decomposer I get the impression that, where heavier types of sludge have to be handled,

this method is better than the Monsanto system. As soon as the organic-material content of the sludge is low enough to prevent excessive dilution of the SO_2 on converting the carbon present in the sludge into carbon dioxide, the second Chemico method based on complete combustion at elevated temperature deserves serious consideration.

In that connexion, I wonder whether anyone here has had experience of the Chemico high-temperature decomposer, it has no mechanical parts, and it seems that it should be very simple. The acid sludge is directly burned in the furnace, and it is doubtful whether there is any advantage in using the low-temperature decomposers, with their necessary burden of mechanical parts, where acid sludges containing little organic material are involved.

THE CHAIRMAN: Mr Nieuwenhuis's description of the difficulties encountered on the acid-sludge recovery plant at Curaçao was of interest to me, as I saw this unit during its period of initial operations. The principle was a new one, based on screw conveyors in place of the rotary furnace. Though theoretically satisfactory, in practice the operators appeared to have had a considerable amount of teething trouble, a not unusual occurrence when starting out under the required difficult operating conditions. It is interesting to note that progress has been made despite the pessimism expressed during the initial operations.

T. H. STOTHARD: I have been wondering whether the Chemico treatment produces such concentration that one product can be reacted with another to give useful chemical products.

THE CHAIRMAN: With regard to the possibility of the oil refiner utilizing his acid sludge for the manufacture of chemical products such as superphosphate, I would refer to the general policy on this question. During my sojourn abroad we had acid sludge available, and a neighbour had locally produced phosphate. The superphosphate possibility was referred back to London, and we were informed that we were in the oil business and not in the chemical business. Nowadays that is not strictly correct, but the policy with regard to superphosphate remains that it is outside the scope of the oil refiner. He is, of course, always willing to dispose of available acid sludge to the superphosphate manufacturer.

A. CLUER: A recent advertisement of the Fluor Corporation in America offers a process for recovery of SO_2 from stack gases, involving absorbing the SO_2 in dimethylaniline. The fact that stack gases are quoted as raw material would imply a low initial SO_2 concentration. The SO_2 may be recovered by this process as liquid, or converted to sulphuric acid by the normal methods.

J. P. K. HAMILTON: It seems to me that here again is a case for the recovery of a valuable product, and that we might be able to obtain an incentive from the Government. I suggest we might give consideration to the problem of obtaining some income-tax relief.

DR J. S. CARTER : A company has been formed to manufacture sulphuric acid from gypsum. One of the products is cement clinker, and there is an association between the sulphuric acid makers and the Associated Portland Cement Manufacturers as to disposal of this.

It would appear that if there were some alliance between the petroleum industry and, say, the National Sulphuric Acid Association it might help.

C. I. KELLY : I understand, rightly or wrongly, from those who handle sludges of various types that, first the acid sludge from white-oil manufacture is very fluid and contains relatively little hydrocarbon and, moreover, it is of the order of 95 per cent strength.

I understand also that if this freshly made sludge is mixed with the acid sludge from heavy oil treatment, which contains something of the order of 40 per cent of acid, a quite fluid material is obtained. It is a question largely of isolating the acid sludges from air and having facilities for mixing them quickly. If that is so, we have a solution of the heavy oil sludge problems associated with the rapid, air-supported, solidification of the hydrocarbon content in heavy lubricating-oil acid sludges.

A. CLUER : Some of the acid sludges from the last stages of production of liquid paraffin, containing 90 to 95 per cent of acid, are used as already indicated. In the material from the stages immediately prior to that the acid percentage is 70 to 80, and we have not as yet found this material reusable; but it is very mobile and will mix with sludges from the earlier stages of oleum treatment and render them more fluid. The mixed sludges will remain fluid and can be transported without any real difficulty. We have had these sludges in tanks in the cold condition for one or two weeks, after which they are still quite pumpable, though the full pump pressure of 400 p.s.i. is then necessary to push the sludge 1400 ft to the boilers. In very cold weather we try to pump the sludge to the boilers while still warm. I do not see why these fluid sludges should not be transported over considerable distances by road or rail car, provided there are precautions against over-heating at the discharging end.

THE CHAIRMAN : The fluidity of acid sludges is a constant problem to the oil refiner. During a visit to the U.S.A. I noted what must be a unique acid sludge. The refinery concerned was treating by the conventional acid method, utilizing agitators, a bright stock from a Mid-West waxy crude. The process was operated at a sufficiently low temperature to precipitate the wax to secure the required pour point on the bright stock. The resultant sludge was thus a mixture of wax and acid sludge. This was run down by gravity from the base of the agitator along an open-top hardwood trough into an open-top paddle mixer; 10 per cent water was added and an emulsion made which was utilized as refinery fuel. This method was stated to be trouble-free. Normally one would expect a solid sludge from bright-stock treatment. Whether or not the presence of wax was responsible for the fluidity of this particular sludge is a point which may be worth consideration.

MR NIEUWENHUIS : I wonder whether the fluidity is the only factor to be considered when trying to convert the sludge into a useful product.

As a matter of fact, it is also the content of organic material in the sludge which seriously complicates the problem. When using a high-temperature decomposer, all the organic material is transformed to CO_2, and this causes a considerable dilution of the SO_2 formed, in the case of sludge rich in carbonaceous material. This means that a very large contact plant would be required for converting the SO_2 into sulphuric acid, which is not very attractive and is uneconomical. When, on the other hand, using a low-temperature decomposer, an excessive production of coke will result, which may be beyond the capacity of the decomposer unit, and probably the handling of the coke will create mechanical difficulties. In addition, with low-temperature decomposition, rather large amounts of cracked hydrocarbons are liberated with the SO_2, making the purification of the SO_2 an awkward problem.

THE CHAIRMAN: What is the minimum acid percentage requirement in the acid sludge to make acid recovery on say a Monsanto unit an economic proposition?

MR NIEUWENHUIS: In the Monsanto type unit as used in Curaçao we could not add more than about 15 per cent of sludges from gasoline refining to the decomposer feed. The percentage of sulphuric acid should be about 80 or higher. It is mainly spent alkylation which is recovered in this unit, and the Monsanto system is probably not applicable for sludges containing a high percentage of organic material.

THE CHAIRMAN: It is encouraging that so much interest has been taken in this very difficult problem of acid-sludge disposal. It has been for years the Cinderella of the oil-refining business. In the time available this evening we may not have been able to produce a golden coach or even these days a chromium-plated one, but I do hope that we have advanced our overall knowledge of this subject, and that the co-operation between the oil refiners so evident this evening may result eventually in some organized form of co-operation.

H. E. F. PRACY: I am very grateful for the privilege of proposing a vote of thanks to Mr Parker for having arranged the symposium and to the authors of the papers presented. As has been said many times this evening, the sludge problem has been the Cinderella of the petroleum industry throughout, and if we are honest with ourselves we must admit that the real reason is that sulphuric acid has always been cheap and in very plentiful supply. We are oil refiners, and the problem has been pushed behind us. Some of us have been fortunate enough to be able to put the sludge into the China Sea, others have burned it under boilers, but have managed to get by with the minimum of trouble, though possibly not with the minimum of annoyance to people working on the plant and living in its vicinity. But I am quite certain that, if we in the petroleum industry devote our attention to the solution of this sludge problem as we have devoted attention during the last few years to many other problems which have arisen, a solution will be forthcoming.

It is very interesting that this evening so many people have gathered

together to discuss this problem, which gives us the hope that there is co-operation between the oil refiners and the plant manufacturers. I hope this chromium-plated or golden coach will appear in the not too distant future.

I ask you to join with me in showing appreciation to those who have made this occasion so successful.

(The vote of thanks was warmly accorded.)

Communication

G. M. Rowell subsequently wrote : It is depressing to note, from a study of the papers presented, that the authors are practically unanimous in recommending disposal, rather than utilization, of acid sludge.

It must be admitted that in the past this has been justified by the poor operating record of nearly every plant built, at least when handling poorer grades of sludge.

The writer believes that the most practicable method is sludge decomposition, and in view of the operating difficulties mentioned by various speakers as being experienced with this type of plant, it may be of interest to mention a new development in this field, the Mathieson–Sasco acid-sludge process, which gives promise of substantially trouble-free operation.

The Mathieson–Sasco process for the recovery of sulphur and coke from acid sludges has been practised on a pilot-plant scale by the Mathieson Chemical Company at Beaumont, Texas. Pilot-plant data now being accumulated at the Beaumont plant lead to the belief that the process is applicable to a large variety of petroleum acid sludges with recovery of 90 per cent of the sulphur probably being feasible. The coke produced is of a dense, hard nature, and is substantially sulphur-free. It leaves the decomposer in a granular form with most of the particles being about ½ inch.

The process is carried out by decomposing the acid-sludge with heat supplied by combustion of part of the coke produced in the process. The remaining coke is withdrawn from the decomposer as a product. SO_2, vaporized hydrocarbons, and products of combustion leave the decomposer as a gas. The gas is cooled by direct contact with cooled recirculated oil, with the consequent condensation of most of the hydrocarbons. The scrubbed gas is fed into a reactor furnace along with air and fuel oil, where a portion of the SO_2 is converted to elemental sulphur and H_2S. The hot gases are cooled by a waste-heat boiler and then passed through one or more catalytic converters with intercooling where further conversion to elemental sulphur occurs.

The equipment comprising the plant and flow through the same are shown in Fig. 1. It may be described as follows :

Acid sludge, which may be from a variety of sources, is accumulated in the sludge-blending tanks where a uniform blend is obtained by recirculation. Blended sludge is fed from one tank at a time into the decomposer. A bed of incandescent coke is maintained in the decomposer by introduction of the proper amount of air which burns a portion of the coke produced. The sludge is fed directly on to the bed of incandescent

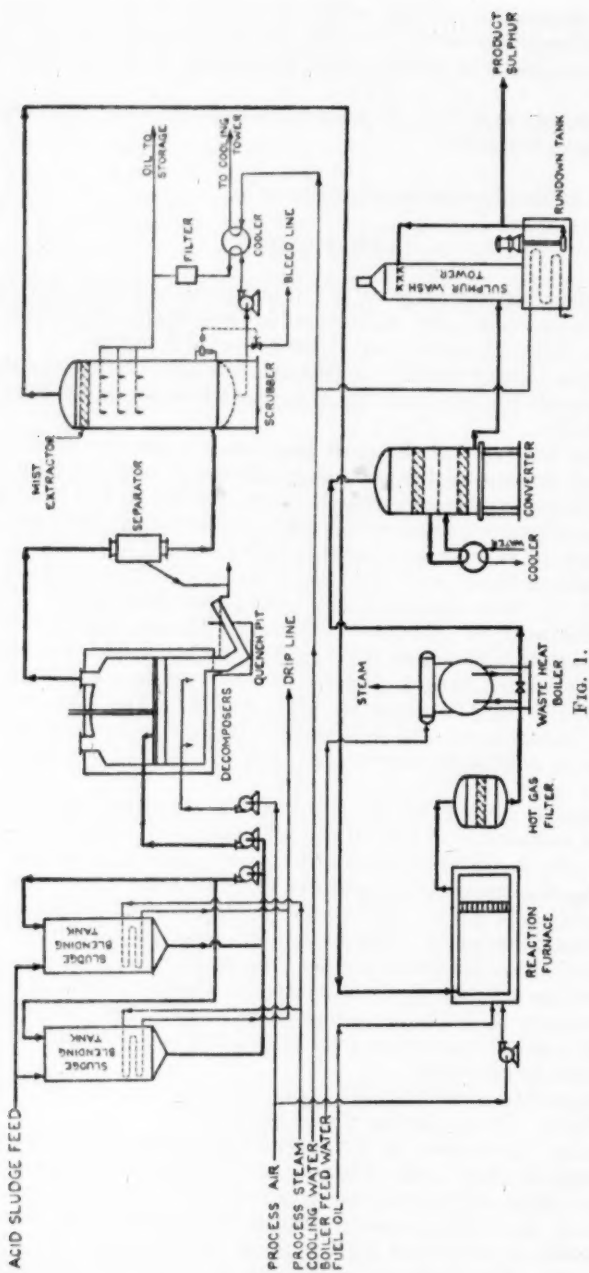

FIG. 1.

FLOW DIAGRAM ACID-SLUDGE PLANT MATHIESON PROCESS

coke with resulting decomposition of the sludge into coke, SO_2, vaporized hydrocarbons, and products of combustion. Coke is withdrawn from the bottom of the decomposer through a quenching pit and is sent to storage.

The SO_2-bearing gas leaves the top of the decomposer at approximately 750° F and is fed to a centrifugal separator for removal of coke breeze. It then passes to a scrubbing tower, where it is cooled by a recirculated stream of oil. This cooling condenses most of the hydrocarbon vapours, and the liquid oil so formed combines with the recirculated stream. The recirculated stream is cooled and filtered for removal of suspended solids. Make oil is withdrawn from the circulating stream to storage

The scrubbed gas, at approximately 200° F, is introduced into the reaction furnace along with air and fuel oil. In this furnace H_2S is formed by the reaction of SO_2 with hydrocarbons from the fuel oil. Some elemental sulphur is also formed in the furnace by reaction of H_2S with SO_2. The reacted mixture is then cooled in the waste-heat boiler, which results in the generation of a considerable quantity of steam. The cooled gases are then sent to one or more catalytic converters, where the H_2S reacts with SO_2 to give additional conversion to elemental sulphur. Intercoolers are used between the catalyst beds for the removal of heat of reaction. Sulphur is condensed from the gas leaving the catalytic converters by direct contact with a recirculated stream of liquid sulphur in the wash tower. The recirculated sulphur is cooled by coils in the sulphur sump. Liquid sulphur product is pumped to liquid storage, or may be solidified by atmospheric cooling in an open pile.

This process from the reaction furnace is similar to the Mathieson–Sasco (modified Claus) process for recovery of sulphur from hydrogen sulphide. A significant difference between the two, however, is that in the acid-sludge process the concentration of sulphur in the gas is appreciably lower, so that it is usually unnecessary to remove sulphur between stages of catalytic conversion.

The catalyst used is bauxite; this does not require reactivation, and has an indefinitely long life. As there is no indirect heating of the coke, and no attempt is made to make sulphuric acid directly from a gas containing oil and water vapours in large amounts, two important sources of trouble have been eliminated.

E. S. Squire and D. G. Pidgeon, in reply to G. M. Rowell, wrote: To the writers' knowledge the subject of acid recovery from acid sludge has been under constant investigation by most of the refiners in the U.K. for the past twenty years, and the objective in holding the symposium was to compare experience. It is therefore a pity that Mr Rowell's written contribution was not offered at the meeting. We have been aware, from the proceedings of the American Chemical Society, of the existence of the Mathieson–Sasco process for some time past, but have been under the impression that the process exists only in the pilot-plant stage. We are, therefore, interested to hear that it is approaching industrial development, and would like to know if Mr Rowell can give figures of results from industrial operation.

DETERMINATION OF OIL CONTENT OF SOLUBLE OIL DISPERSIONS

By Cutting Oils Panel of Standardization Sub-Committee No. 6—Lubricants

Summary

The Cutting Oils Panel has submitted a method for the determination of the oil content of soluble oil dispersions. The work of the Panel in developing this method is described in detail. The method consists of splitting a known volume of soluble oil dispersion with 20 per cent hydrochloric acid in a graduated flask and measuring the volume of oil liberated. Several alternative splitting agents were investigated and rejected.

Early in 1949 the Cutting Oils Panel decided to investigate tests for the determination of the oil content of soluble oil dispersions, a determination which is widely used both in laboratory and workshop, and it was agreed that standardization of a simple text should be undertaken. In outline, the methods already in use involve treatment of the dispersion with an acid splitting agent followed by volumetric determination of the separated oil. The reaction is generally carried out in a flask with a graduated neck, the separated oil stratum being brought into the neck for measurement by further addition of splitting agent.

The early stages of the work were restricted to comparing the relative merits of hydrochloric acid and sulphuric acid as splitting agents with used and unused soluble oil dispersions. Details of the test method and the recorded values are shown in Appendix I. Results obtained with both splitting agents were similar, but hydrochloric acid was selected as being more readily available and simpler in handling. It was also noted at this stage that the separated oil stratum obtained with used dispersions often contained solid matter and that clear boundaries with the aqueous solution and the glass were not obtained.

Two further series of tests were made using a method based on the experience gained during the first series. A variety of used and unused dispersions were examined, the range of original oil content covered being up to 5 per cent. The dispersions examined were prepared from both " clear " and " milky " types of soluble oil.

The results obtained in series II showed that for the lower concentrations the values obtained were not sufficiently precise. Accordingly, in series III, modification of the test method was made to allow a larger sample to be used for dispersions of low concentration. At the same time a procedure was evolved for sampling a dispersion in which separated oil was present, the test then being made on a uniform dispersion. The details of the experimental work involved are shown in Appendixes II and III.

Although the experience thus gained gave a sound working basis for a standardized method, it was deemed advisable to widen the scope of investigations and to consider the use of other splitting agents. These were

known to be used industrially, and were claimed to be superior to hydrochloric acid. A particular merit claimed for some reagents was a high rate of oil separation enabling results to be reported within a few hours and in some cases by cold separation. A survey was accordingly made using the following reagents :—

(*a*) Copper sulphate—a saturated solution in 2 per cent (v/v) aqueous sulphuric acid.
(*b*) Barium chloride—20 per cent solution in 10 per cent (v/v) aqueous acetic acid.
(*c*) Barium chloride—20 per cent solution in 10 per cent (v/v) aqueous hydrochloric acid.
(*d*) Aluminium nitrate—10 per cent solution in 10 per cent (v/v) aqueous nitric acid.
(*e*) Ammonium sulphate—saturated aqueous solution.
(*f*) Hydrochloric acid—20 per cent (v/v) aqueous solution.

The details of the experimental work are recorded in Appendix IV.

It was found that although some of the reagents mentioned gave a slightly more rapid rate of oil separation than 20 per cent hydrochloric acid they caused other undesirable effects. Those based on barium and aluminium separated flocculent, insoluble materials which caused extensive interfacial "cuffs." The barium solutions, moreover, yielded oil layers which gelled on cooling. It was again generally noted that the used dispersions caused the greatest separation of flocculent material. Copper sulphate solution gave rapid separation at room temperature with three out of the four dispersions tested, and thus might be more applicable than 20 per cent hydrochloric acid, which required heating for effective separation. Concentrated hydrochloric acid as a primary splitting agent was also suggested for use at room temperature.

Accordingly, the next investigation was a close comparison of the copper sulphate and the hydrochloric acid methods. In all, seven samples were co-operatively examined, including used and unused dispersions, some of the latter containing sodium carbonate added as a corrosion inhibitor. The range of oil contents taken was representative of those found in practice.

It was found generally that the copper sulphate solution, when used at room temperature, failed to give rapid separation with the majority of dispersions tested. Therefore, the results reported were obtained by heating the solutions in the normal manner.

Concentrated hydrochloric acid was equally ineffective at room temperature, four out of nine dispersions tested failing to separate in the cold after 30 minutes standing. The results reported are, therefore, those obtained by hot separation.

The results of this investigation are given in Appendix V. Close observation of the rate of separation of the oil stratum was made by certain laboratories and typical examples are shown in Figs. 1 to 4.

The results clearly demonstrated that, for the majority of cases, 20 per cent hydrochloric acid was a reliable reagent. There appeared to be no general advantage which would warrant the adoption of either copper sulphate solution or concentrated hydrochloric acid as a splitting agent.

Accordingly, a method based on the use of 20 per cent hydrochloric acid was prepared by the panel, and has been submitted to the Standardization Committee for approval. This method is detailed in Appendix VI.

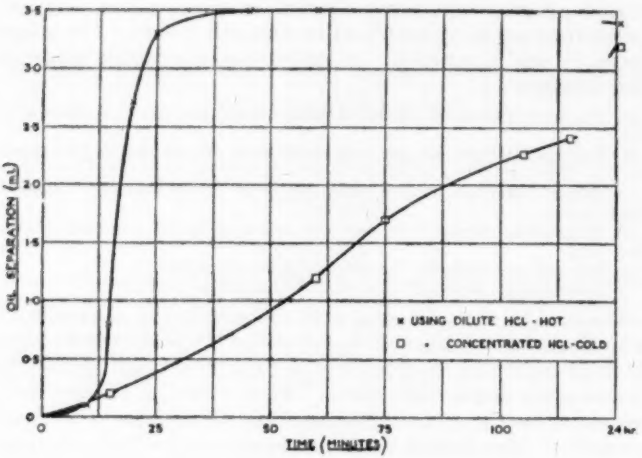

FIG. 1

RATE OF OIL SEPARATION—DISPERSION P

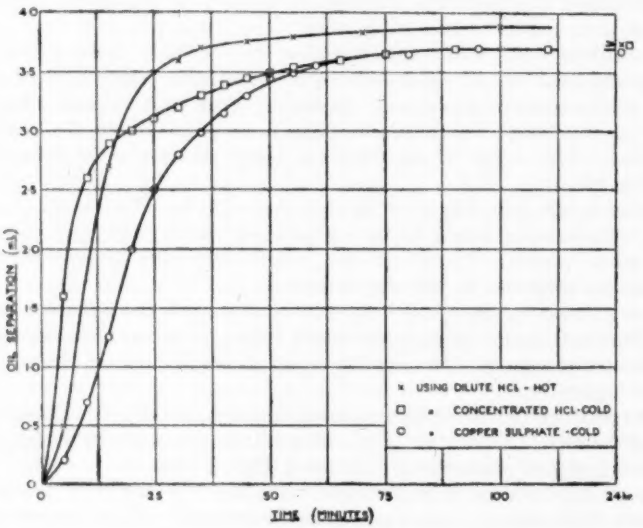

FIG. 2

RATE OF OIL SEPARATION–DISPERSION R

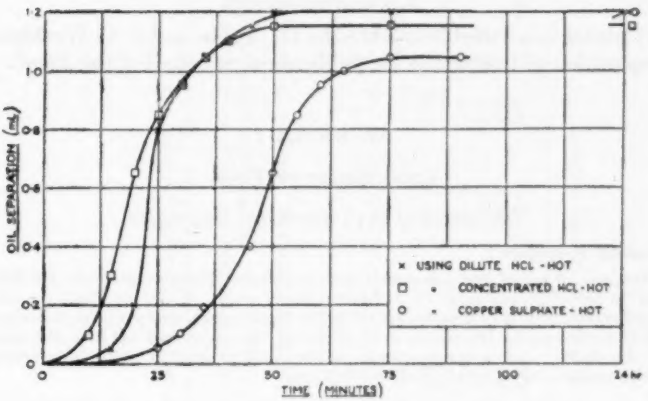

FIG. 3

RATE OF OIL SEPARATION—DISPERSION U

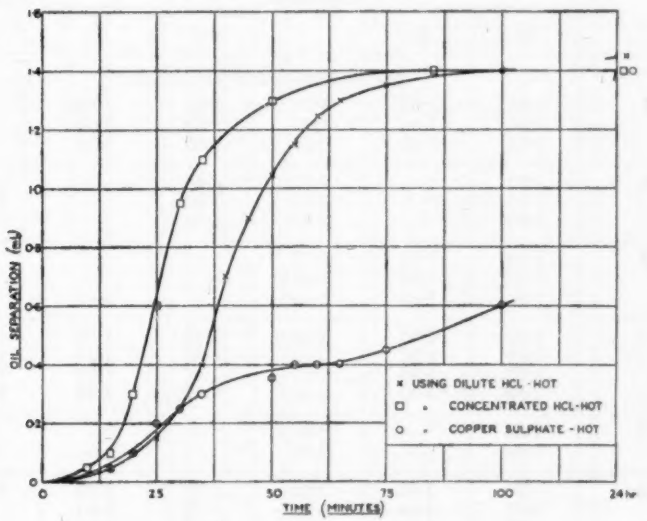

FIG. 4

RATE OF OIL SEPARATION—DISPERSION V

ACKNOWLEDGMENT

The Cutting Oils Panel is indebted to H. Crabbe and C. C. Washbrook for the preparation of this article for publication on behalf of the Panel.

APPENDIX I

FIRST SERIES OF TESTS

Oil Content of 25 : 1 Soluble Oil Dispersions

Experimental Procedure :

(1) Measure 50 ml of the dispersion into a 150-ml Hirschsohn flask (BS 676/1936) and add 20 ml of 20 per cent (v/v) hydrochloric acid. Heat the flask on the water bath until separation is complete, swirling the flask occasionally to aid the separation.

(2) Add sufficient hydrochloric acid to bring the separated oil into the graduated neck of the flask. Allow the contents of the flask to cool to room temperature and record the volume of oil to the nearest 0·05 ml.

Laboratory	Hydrochloric acid			Sulphuric acid		
	A. Unused milky dispersion		B. Used dispersion	A. Unused milky dispersion		B. Used dispersion
	(*a*)	(*b*)		(*a*)	(*b*)	
	Oil content—ml/50 ml sample					
1	1·85	1·85	1·75	1·85	1·85	1·75
	1·85	1·80	1·75	1·85	1·85	1·75
	1·85	1·85	1·75	1·85	1·85	1·75
2	1·80	—	1·70	1·80	—	1·70
	1·80	—	1·70	1·80	—	1·70
	1·80	—	1·70	1·80	—	1·70
3	1·80	1·75	1·75	—	—	—
	1·80	1·80	1·80	—	—	—
	1·80	1·80	1·75	—	—	—
4	1·92	1·96	1·83	1·93	1·95	1·87
	1·88	1·97	1·87	1·89	1·94	1·84
	1·93	1·94	1·87	1·92	1·93	1·87
	1·89	1·95	1·86	1·92	1·96	1·85
5	1·90	—	1·80	—	—	—
	1·90	—	1·80	—	—	—
6	1·79	1·65	1·70	1·75	1·63	1·67
	1·77	1·65	1·70	1·73	1·64	*
	1·80	1·68	1·70	1·77	1·62	*
	1·79	1·66	1·70	1·75	1·63	*
7	1·90	1·85	1·85	1·90	1·90	1·85
	1·90	1·90	1·80	1·85	1·90	1·85
	1·95	2·00	1·95	1·85	1·90	1·85

Notes : (*a*) Samples of the same emulsion.
(*b*) Samples of emulsions prepared by the laboratory in question, from the same soluble oil.
* Too opaque to read.

APPENDIX II

Second Series of Tests

Oil Content of Various Soluble Oil Dispersions

Experimental Procedure :

(*a*) To 50 ml of the sample in the 150-ml graduated flask (BS 576/1936) add 20 ml of 20 per cent (v/v) hydrochloric acid.

Note 1 : If free oil is observed on the surface of the original sample avoid inclusion of this free oil by pipetting 50 ml of liquid from below the surface.

(*b*) Heat on a steam-bath until the separation of oil is observed to be complete, swirling the flask from time to time so as to detach any oil adhering to the sides of the flask. Add sufficient 20 per cent hydrochloric acid to bring the separated oil into the graduated neck of the flask.

(*c*) Allow to cool to room temperature and adjust the oil level if necessary by further addition of the dilute acid. Record the volume of oil to the nearest 0·05 ml.

Laboratory	1			2			3			4		
	Oil content—ml/50 ml sample											
C. Unused dispersion .	4·3	4·35	4·35	4·45	4·4	4·4	—	4·35	—	4·35	4·35	4·4
D. Unused dispersion .	2·1	2·0	2·05	2·15	2·15	2·10	—	2·25	—	1·9	1·95	1·95
E. Unused dispersion .	0·8	0·8	0·8	0·9	0·85	0·9	—	0·9	—	0·8	0·8	0·8
F. Unused dispersion .	1·9	1·9	1·9	1·95	1·9	1·95	1·85	1·95	1·90	1·8	1·8	1·8
G. Used dispersion .	2·7	2·6	2·6	2·9	2·9	2·95	2·95	2·9	—	2·9	2·9	—
H. Used dispersion .	3·7	3·55	3·6	3·75	3·7	3·7	—	3·65	—	3·65	3·7	3·8

Notes : (1) In the case of dispersion D the demarcation between oil and aqueous phase was poorly defined.
(2) There was considerable " tailing " with the used dispersions.

APPENDIX III

Third Series of Tests

Oil Content of Soluble Oil Dispersions

Experimental Procedure :

(*a*) Gently shake the sample of soluble oil dispersion sufficiently to make uniform, pour into a separating funnel, and allow to stand overnight. With samples of not more than 500 ml pour the whole of the sample into a funnel of suitable size. With larger samples take approximately 500 ml.

(*b*) After standing overnight, run off all the dispersion, including any "cream," leaving only free oil in the funnel, and stir the dispersion to make uniform.

(*c*) Measure 100 ml of the dispersion into a graduated 150-ml flask (BS 676/1936) and add 20 ml of 20 per cent (v/v) hydrochloric acid.

(*d*) Heat on a steam-bath until the separation of oil is observed to be complete, swirling the flask from time to time so as to detach any oil adhering to the sides of the flask. Add sufficient of 20 per cent hydrochloric acid to bring the separated oil into the graduated neck of the flask and allow the flask to stand overnight at room temperature.

(*e*) Adjust the oil level, if necessary, by further addition of dilute acid. Record the volume of oil to the nearest 0·05 ml.

Laboratory	Oil J. Unused milky dispersion			Oil K. Unused clear dispersion		
	25 : 1	50 : 1	100 : 1	25 : 1	50 : 1	100 : 1
	Oil content—ml/50 ml sample					
1	3·15 3·20	1·60 1·70	0·75 0·80	3·20 3·05	1·60 1·60	0·80 0·80
2	3·20 3·20	1·70 1·70	0·75 0·85	3·05 3·05	1·60 1·60	0·80 0·80
3	3·30 3·30	1·70 1·70	0·85 0·85	3·10 3·10	1·65 1·60	0·85 0·80
4	3·20 3·25	1·65 1·60	0·80 0·80	3·00 3·05	1·55 1·60	0·80 0·70
5	3·25 3·27	1·68 1·72	0·92 0·85	3·21 3·12	1·75 1·75	0·87 0·88
6	3·25 3·27	1·70 1·73	0·88 0·90	3·10 3·10	1·68 1·68	0·82 0·82
7	3·20 3·20	1·70 1·70	0·90 0·90	3·10 3·10	1·70 1·70	0·90 0·85
8 *	3·28 3·35	1·75 1·75	0·88 0·93	3·20 3·15	1·74 1·75	0·88 0·86
9	3·20 3·20	1·60 1·60	0·80 0·80	3·0 3·0	1·65 1·65	0·90 0·90
10	— —	1·75 1·75	— —	3·05 3·10	1·75 1·75	0·80 0·80

Note : * In this laboratory the top meniscus was read in each case.

APPENDIX IV

Fourth Series of Tests

Determination of Oil Content of Soluble Oil Dispersions Using Various Reagents

Experimental Procedure :

Working details as in Appendix III, but substituting the selected reagent for 20 per cent (v/v) hydrochloric acid where indicated.

Table I

Oil Content per cent by Volume

Reagent	Laboratory	L Unused dispersion	M Unused dispersion	N Unused dispersion	O Used dispersion
20% hydrochloric acid	1	8·6	4·1	1·7	3·2
	2	9·0	4·1	1·85	3·3
	3	8·4	3·9	1·65	2·9
	4	8·5	4·1	1·8	3·2
	5	8·2	4·1	1·8	3·2
	6	8·3	3·9	1·7	3·2
	7	8·6	4·1	1·7	3·2
	8	8·2	4·0	1·7	3·6
Aluminium nitrate/nitric acid	2	9·2	4·1	1·85	3·75
	3	8·6	3·8	1·7	2·7
Barium chloride/acetic acid	4	8·25	3·9	1·6	3·2
	5	8·25	3·8	1·6	*
Barium chloride/hydrochloric acid	4	8·5	4·1	1·8	3·2
	5	8·2	3·9	1·6	*
Copper sulphate/sulphuric acid	6	8·4	3·8	1·6	3·2
	7	8·5	4·2	1·6	*
Ammonium sulphate/ solution	8	8·6	3·6	1·6	3·1
	1	8·5	4·05	1·6	*

Note : * In these cases oil separation was incomplete and gave poorly defined boundaries preventing accurate reading of the volume.

APPENDIX V

Fifth Series of Tests

Oil Content of Used and Unused Soluble Oil Dispersions—Comparison of the Use of Hydrochloric Acid and Copper Sulphate Solutions

Reagent	Copper sulphate solution						Hydrochloric acid, 20%						Hydrochloric acid, conc				
Laboratory :	1	2	3	4	5	6	1	2	3	4	5	6	2	3	4	5	6
P. Unused milky dispersion .	3·25 *	3·90	3·30	3·00	3·00	*, ¶	3·15 *	3·50	3·05	3·25	3·00	3·25	3·40	3·30	3·00	3·00	2·90
R. Unused milky dispersion .	3·75	3·60	3·85	3·70	3·55	3·90	3·75	3·70	3·85	3·75	3·60	3·85	3·70	3·80	3·75	3·60	3·90
S. Used dispersion . .	7·35 *	7·40	7·25	7·20	6·60	7·05	7·15 *	—	7·25	7·20	6·60	6·70	—	7·00	7·00	6·65	7·00
T. Used dispersion . .	4·85 †	4·70	4·90	4·35	4·30	4·65	4·90 †	4·70	4·40	4·95	4·30	4·30	4·70	4·55	4·25	4·25	4·20
U. Used dispersion . .	1·30 †	1·20	1·25	1·20	1·10	1·00	1·25 †	1·20	1·20	1·20	1·10	1·00	1·30	1·25	1·15	1·10	1·05
V. Used dispersion . .	1·50 *	1·30	1·25	1·45	1·45	1·40	1·50	1·50	1·45	1·55	1·50	1·40	1·55	1·50	1·40	1·50	1·45
W. Used dispersion . .	2·80 †	‡, ¶	2·20	—	‡, ¶	†, ‡	2·30	2·90	2·55	2·70	2·30	2·40	3·10	2·70	2·60	2·30	2·80
X. Used dispersion . .	4·80 *	4·80	5·10	4·15	4·25	†, ¶	4·40 *	5·00	4·10	5·10	4·25	4·20	4·80	5·05	4·10	4·30	3·90
Y. Used dispersion . .	—	1·00	1·00	1·25	—	1·20	—	0·90	1·05	1·05	0·70	—	1·00	—	1·00	—	1·00

Notes : * Much solid matter separated, but reading not difficult.
† Much solid matter separated, rendering reading difficult.
‡ Carbon dioxide evolved freely.
¶ Reading unobtainable.

APPENDIX VI

Soluble Cutting Oil

Oil Content of Dispersions

IP 137/52

Introduction

1. (*a*) This method determines the oil content of unused and used soluble cutting-oil dispersions.

(*b*) The *oil content* is defined as the amount of material which separates under the conditions of this test as an oily layer insoluble in the aqueous phase. In addition to the mineral oil originally present in the soluble oil, the separated material may also include portions of those emulsifiers which are decomposed by the acid treatment.

(*c*) In order to relate the results of this test to the actual content of soluble oil in the dispersion, a similar test should be made on a dispersion of the given soluble oil which has been accurately diluted to a comparable extent.

Outline of Method

2. A known volume of the soluble cutting oil dispersion is treated with hydrochloric acid in a graduated flask and the volume of liberated oil is measured.

Apparatus

3. (*a*) *Separating Funnel*—of 500 ml capacity or less according to the amount of sample available.

(*b*) *Flask*—of 150 ml capacity and with a 10-ml graduated neck, conforming to BS 676/1936.

Materials

4. *Hydrochloric Acid*—20 per cent (v/v).

Preparation of Sample

5. (*a*) Shake the sample gently sufficiently to make it uniform. Pour 500 ml of the sample, or the whole of the sample if less than 500 ml, into a separating funnel of an appropriate size, and allow it to stand overnight.

(*b*) After allowing the sample to stand overnight, run off all of the dispersion, including any creamy layer, into a suitable beaker, leaving only free oil in the funnel. Stir the dispersion to make it uniform and test it without further delay.

Procedure

6. Measure 100 ml of the uniform dispersion from a 100-ml measuring cylinder into the graduated flask and add 40 ml of the hydrochloric acid. When the dispersion is known to have an oil content of more than 10 per cent, or when a volume of oil of more than 10 ml is found to separate as a result of the procedures described in Sections 6 (*b*) and (*c*), measure a smaller quantity of the dispersion such as to give a convenient volume of separated oil; add sufficient hydrochloric acid to give a total volume of approximately 140 ml.

(*b*) Swirl the flask until its contents are thoroughly mixed and heat it in a boiling water-bath for 1 hr. Remove the flask from the bath and fill it to the top graduation with the hydrochloric acid. Allow the flask to stand for 1 hr, swirling it from time to time to detach any oil adhering to the sides.

(*c*) Adjust the oil level if necessary by further addition of the hydrochloric acid. Determine the difference in the scale readings corresponding with the top edge of the meniscus at the upper and lower levels of the separated oil and record the volume to the nearest 0·05 ml.

Calculation and Reporting

7. Calculate the result as a percentage by volume and report it to the nearest 0·1 per cent as the Oil Content, IP 137.

Precision

8. (*a*) For unused soluble-oil dispersions duplicate results should not differ by more than the following amounts :

Repeatability	Reproducibility
0·1%	0·2%

(*b*) With used soluble-oil dispersions difficulty may be experienced in reading the volume of separated oil, depending on the condition of the used dispersion. For this reason no acceptable limits can be given.

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Oilfield Exploration and Exploitation

Geology

1. The significance of populations in palæontology and stratigraphy. H. Hiltermann. *Erdöl u. Kohle*, 1951, **4** (5), 244–9.—There is in palæontology too great a multiplication of specific names as a result of excessive "splitting," and this leads to unwieldy classifications sometimes hardly understood even by the expert. Author makes a strong plea for the detailed study of fossil populations or communities, rather than of

isolated members of such communities, according to modern taxonomic principles and such unifying concepts as palæo-ecology. Published work embodying this trend is discussed. S. E. C.

2. Contribution to the problem of origin of ground waters. Z. Obuchowicz; Notes on paper by Z. Obuchowicz. W. Chajec. *Nafta*, Aug. 1950, **6**, 216–20.—Referring to a paper entitled "Iodine and Bromine in deep oil well brines," by W. Chajec (*Nafta*, Dec. 1949, **5**, 366–72), Z. Obuchowicz discusses the use of ionic ratios as a factor for determination of age of any enclosed volume of water. Quoting A. Lane, L. S. Balashov, and others he discusses the choice of actual ions which may give suitable results, expressing himself in favour of $Br' : Cl'$ or $Ca'' : Sr''$ instead of $Na' : Cl'$. Five tables are included to prove this point. W. Chajec in reply states that $Na' : Cl'$ ratio may still be used, but with reference to Polish underground waters the lowest critical value is not 0·55 but 0·8 to 0·9. Other ionic ratios must not be left out of determination of the origin of such waters. M. S.

3. Tectonic divisions of North America. A. J. Eardley. *Bull. Amer. Ass. Petrol. Geol.*, 1951, **35** (10), 2229.—Review of the tectonic framework of North America presented as one of the Distinguished Lectures of the American Association of Petroleum Geologists in 1951. The theories expressed, although not in accord with the opinions of some geologists, reflect modern thought and seem to be the best documented and most acceptable to date.

Author divides North America into the following sections: the Canadian shield, the Greenland shield, the central stable region, the Arctic stable region, the orogenic belts of the Atlantic margin, the orogenic belts of the Pacific margin, the orogenic belts of the Rocky Mountains, and the Coastal Plains, and concludes with a review of the pre-Cambrian orogeny of the Canadian shield, which is the great stable part of the North American continent, continuous with the Greenland shield, and one of several great shields nearly surrounding the Arctic Sea. From a study of the shields and associated belts and basins, it appears that Eurasia and North America were broadly connected over the north polar region in early Palæozoic time; and that a once-great shield common to all the continental crust of the northern hemisphere has been broken by orogenic belts within it. E. N. T.

4. Western Kansas' Mississippian. P. C. Ingalls. *Oil Gas J.*, 27.9.51, **50** (21), 137.—The oil possibilities in the little tested Mississippian rocks lying on an erosional shelf on the eastern limb of the Hugoton embayment of the Anadarko Basin are believed to be greater than previously supposed. Although the tests made here have been mainly unsuccessful it is considered that production may be found similar to the "spotty" development on the Arbuckle arch, since oil may have migrated from the Pennsylvanian to the underlying Mississippian rocks and accumulated in porous zones along the Mississippian–Pennsylvanian (Marmaton) unconformity. C. A. F.

5. Eastern Kansas development and prospects. J. M. Jewett. *World Oil*, Aug. 1951, **133** (3), 100.—Recent important oil discoveries in Eastern Kansas are described. These include the Davis Ranch, Newbury, Bushong, and Sabetha fields in which the pays are Pennsylvanian, Mississippian, Simpson, Hunton, and Viola limestones and sands.

The Abilene Arch area to the west of the Nemaha anticline and the western flank of the Nemaha Uplift have been inadequately tested. The importance of the general structural pattern of the area and local variations within it are emphasized.

Maps showing the location of important recent tests in Eastern Kansas and structural trends are included. Six references are given. C. A. F.

6. Don't sell northeast Texas short. P. C. Ingalls. *Oil Gas J.*, 4.10.51, **50** (22), 347.—Although no important fields have been discovered in East Texas in recent years in spite of continued wildcatting, the prospects of the area may be greater than is currently believed.

Of sixteen shallow piercement domes which have been discovered around the axis of the basin, only one has been found to hold commercial oil, and the remainder have not been tested in detail. The Jurassic Smackover limestone, which is porous to the

east, where it is producing in Louisiana and Kansas, has not been tested within the basin, and it may have been uplifted here by salt movements. C. A. F.

7. The Spraberry play in West Texas. R. C. Senning. *World Oil*, Sept. 1951, **133** (4), 125.—Since the discovery of commercial oil in the Lower Permian Spraberry sand in the Midland Basin of West Texas in Jan. 1949, 2,903,155 brl had been produced by the end of May 1951 from 248 wells. There is considerable leasing and wildcatting in the area, which has become an important source of high-gravity oil.

The Spraberry, which varies from 1520 to 935 ft in thickness and is divided into three sand bodies, is restricted to the Midland Basin area of the Permian Basin. To the west and south the sand grades into shale and undifferentiated sands along the edges of the basin, and to the north and east into limestones and shales. Oil accumulation in the Spraberry is controlled by two structural conditions: structural relief above deep-seated Pennsylvanian highs and strike stratigraphic trapping on a broad monocline. There is complex vertical fracturing in the sand, probably due to shearing.

Pipelines in the area and reservoir data are briefly discussed. A cross-section, typical electric log, and structure map are included. C. A. F.

8. Uinta Basin's activity climbs. P. C. Ingalls. *Oil Gas J.*, 20.9.51, **50** (20), 357.—Three fields have been discovered in the Tertiary Uinta Basin in northwestern Utah since 1949. These are the Roosevelt, Red Wash, and Duchesne fields. Roosevelt and Red Wash produce from the Green River, and Duchesne from the Tertiary Wasatch. Prior to the finding of these fields, oil possibilities in the continental sediments of the basin were considered poor. The recent discoveries indicate the reserves of the area to be considerable. C. A. F.

9. Williston Basin oil possibilities. G. W. Smith. *World Oil*, 1.7.51, **133** (1), 78.—Since the recent discovery of commercial oil in the Williston Basin by the well, 1 Iverson in North Dakota, there has been intensive leasing and exploration activity in the area. The discovery well flowed approx 400 b.d. of 47° to 53° oil from 11,630 to 11,660 ft from Middle Devonian.

Colorado shales, marine Jurassic, Eaglewood bituminous shales, and marine sections in the Mississippian and Palæozoic are possible source rocks in the basin, and possible reservoir rocks include sands in the Muddy and Dakota and porous limestones in the Madison and Devonian. Possible structures include the Nisson anticline and the Keene dome, lenticular traps in the Dakota and Muddy, truncation and pinchout traps in the Palæozoic along the eastern and southern flanks of the basin, and unconformity traps at the base of a number of formations.

A contour map of the base of the Colorado group and a diagrammatic cross-section of the Williston Basin are included. C. A. F.

10. Williston well finds oil. Anon. *Oil Gas J.*, 11.10.51, **50** (23), 84.—1 Pine Unit, Wibaux County, Montana, recovered on test 370 ft of 29·4° oil and 180 ft of gas cut mud from 8924 to 8975 ft. Pay is probably Lower Devonian or Upper Ordovician. C. A. F.

11. Palæozoic stratigraphy of southwestern Montana. L. L. Sloss and C. A. Moritz. *Bull. Amer. Ass. Petrol. Geol.*, 1951, **35** (10), 2135.—All Palæozoic systems except the Silurian are represented in the southwestern Montana area. Cambrian strata are similar to those of better-known areas on the north and east, but differ in the presence of several intrasystemic unconformities; the Ordovician Kinnikinic quartzite occurs along the Idaho–Montana border, and a thin edge of Bighorn dolomite is present along the eastern margin of the area, in Gallatin County. In other areas the Cambrian strata are overlain by Upper Devonian.

In the Jefferson and Three Forks formations the Devonian strata are typically developed and widespread, with little lateral variation, but the Mississippian system is characterized by extremes of facies expression, including rapid lateral variation among redbeds, evaporites, fragmental limestones, dolomites, and black shales. Mississippian rocks range in thickness from 1000 ft to more than 4000 ft.

The quartzose and well-cemented sandstones of the Quadrant formation, ranging in thickness from about 100 ft to almost 3000 ft, dominate the Pennsylvanian strata.

The Permian system is represented by the Phosphoria formation, which is composed of quartzites, limestones, dolomites, black shales, phosphorites, and bedded cherts. E. N. T.

12. Colorado shale of central and northwestern Montana and equivalent rocks of Black Hills. W. A. Cobban. *Bull. Amer. Ass. Petrol. Geol.*, 1951, **35** (10), 2170.—The Colorado shale of central and northwestern Colorado and the equivalent rocks on the north flank of the Black Hills are dominantly dark-grey marine shales. Though the Colorado shale has commonly been treated as a single formation, with little attempt at subdivision, while the equivalent rocks of the Black Hills have been divided into eight formations and several members, closer examination of the Colorado shale shows that it too is made up of many distinct lithological units, most of which can be correlated with the named units of the Black Hills.

The formations in the Black Hills which are equivalent to the Colorado shale are the Fall River sandstone, Skull Creek shale, Newcastle sandstone, Mowry shale, Belle Fourche shale, Greenhorn formation, Carlile shale, and Niobrara formation. Taking the features of the shale on the Mosby dome of the Cat Creek anticline in Petroleum and Garfield Counties as typical for central Montana, and the Sweetgrass arch as typical for northwestern Montana, each formation of the Colorado shale and its equivalent part of the section of the north flank of the Black Hills are briefly described, compared, and contrasted, their fossil contents listed, and the more important guide fossils pointed out. E. N. T.

13. Significant developments in the Four Corners area. J. L. Tatum. *World Oil*, 1.7.51, **133** (1), 73.—There have been a number of important oil developments in the Four Corners area in the Rocky Mountain region during the last two years. These include the discovery of an oil-bearing reef at Dove Creek and rapid extension of the gas-producing area in the San Juan Basin.

There are three major sedimentary basins in this area—the San Juan, Black Mesa, and Paradox basins. Much evidence has been found of Palæozoic structural movements in these basins which was previously suspect.

An isopach map of the Pennsylvanian, a location map, and a chart showing well completions in the three basins since 1946 are included. C. A. F.

14. Relationship of hydrocarbon gravities to facies in Gulf Coast. F. R. Haeberle. *Bull. Amer. Ass. Petrol. Geol.*, 1951, **35**, 10, 2238.—The structural arrangement of the wedge-shaped mass of sediments in the Gulf Coast, thickening and dipping towards the Gulf of Mexico, presents an ideal situation for the study of the relationship of the geologic age, depth of burial, and composition and gravity of hydrocarbons; it is possible to encounter producing zones of different geologic ages at the same depth by moving inland and thus updip. Since the original theory that the gravity of hydrocarbons is controlled by their geologic age and depth of burial was advanced in the 1930s, very little further work has been done until recently on the problem of the varying gravities of hydrocarbons on the Gulf Coast, despite the fact that the majority of the deep wells in this region were not drilled until after 1938. Recent studies have shown that while changes in gravity of hydrocarbons may exist in older and deeper zones, these changes are more closely related to the action of catalysts present in clays than to the influence of depth and pressure; and that the appearance of higher-gravity hydrocarbons in deeper zones may be the result of their presence in the more marine shaly facies of the various formations rather than to their presence in older and deeper zones.

Average gravity figures in the Gulf Coast show significant variations from the original theory and illustrate the relationship between the occurrence of more marine conditions and higher-gravity hydrocarbons. E. N. T.

15. Oölitic iron deposits of New Mexico. V. C. Kelley. *Bull. Amer. Ass. Petrol. Geol.*, 1951, **35** (10), 2199.—During investigations of iron ore in New Mexico it was found that in widely separated areas the lower part of the Bliss sandstone, which is Upper Cambrian in age, contains beds of oölitic hematite. In previous studies of the Bliss sandstone these beds have been entirely overlooked, but in the present survey, although of wide reconnaissance, local sections of the Bliss sandstone have been measured in detail. Many chip samples of the oölitic hematite have been assayed, and reserves

and tenor of the low-grade ores have been estimated. Thin sections and polished sections of the samples from most of the deposits in southwestern New Mexico have been studied.

The beds of oölitic hematite have been found to be commonly as much as 15 ft in thickness, and are exposed in outcrops as much as several miles in length in the Caballe, San Andres, Black, and Silver City mountains. The oölitic ores appear to be associated with the calcareous, gautonitic, and hematitic facies of the sandstone, and it is concluded that the iron content of the deposits is almost entirely syngenetic and that deposition may have been mechanical as well as chemical. Much of the hematite has been diagenetically rearranged and considerably recrystallized, with the result that the oölites are in various stages of obliteration, with many oölites entirely obliterated. E. N. T.

16. A look at Northern Alberta. P. C. Ingalls. *Oil Gas J.*, 11.10.51, **50** (23), 165.—Limited exploration work has been carried out in recent years in the Peace River area and, although no major discoveries have been made, the area has good prospects.

Large gas wells have been completed in Lower Cretaceous, Triassic, and Permo-Pennsylvanian rocks in the area, and oil production has been found at Normandville and Belloy.

Eastward there is rapid thinning of the Mesozoic, and there may be good prospects in eastward pinchout structures.

To the east of the Peace River area are the Athabaska tar sands, the oil content of which is currently estimated at 100 to 250 billion brl. Pilot operations have indicated that the oil can be produced from these sands, possibly competitively. C. A. F.

17. Alberta joint venture results in 675-barrel well. Anon. *World Oil*, Sept. 1951, **133** (4), 334.—Wagner-Matlo-Dillabough-Ad Astra 3 in the Campbell area, Alberta, initially produced on test approx 675 b.d. C. A. F.

18. Study of the Central Cordillera in the Huesca region. A. Alemala. *Bull. Ass. franç. Tech. Pétrole*, 31.7.51 (88), 30–8.—The Trias of the Huesca region has variegated marls and fetid limestones. There is gypsum in the marls. Some of the limestone is clearly younger than the Keuper despite its resemblance to the Muschelkalk, but there are fetid Keuper limestones and perhaps even Muschelkalk. South of Liguerre de Cinea there are bitumen-impregnated limestones, and some years ago attempts were made to exploit them. West of the Noguera Robagorzona River the Lias and Jurassic disappear, and in Haut Aragon there is a break from Trias to Upper Cretaceous. The Cenomanian and even the Senonian rest on supra-Keuper fetid limestones. In the Sierra de Estadilla the Senonian, 300 m thick, consists of compact fractured limestones. In the lower parts the limestones are sandy. 50 m of red marls with calcareous sandstone in the Sierra de Guara may be overlain by Maestrichtian. The Senonian is thin in the Sierra de Santo Domingo. The Eocene is less thick than farther north, and consists of fine limestone, hard sandstones, and sandy marls with *Alveolina*, *Nummulates*, *Assilina*, etc., followed by Lutetian marls. The marls pass laterally and upwards into a flysch facies. Some continental rock types are found in the flysch in the Castillo de Fantoba zone. The flysch is 600 to 1100 m thick. Continental deposits overlie the flysch, and at Grauss Oligocene conglomerates rest discordantly on the flysch. Lacustrine deposits have been recognized. Presumed Miocene conglomerates have been observed.

The Sierras clearly show two fold systems which are substantially at right-angles. One system runs north–south; its age is pre-Ludian.

The Eocene has gas seeps north of the asphaltic limestones of Liguerza. The Lacq oilfield lies still farther north. The Campodarbe and Bínces anticlines seem to have cores of Lower Eocene, and hence the Cretaceous may be shallow, but it differs from the French Cretaceous. G. D. H.

19. Germany has gasser. Anon. *World Oil*, Aug. 1951, **133** (3), 280.—Frenswegen 1, north of Nordhorn near the Dutch–German border, found commercial gas at 6316 ft in Zechstein Plate dolomite, which is also the pay in the Bentheim field. C. A. F.

20. German petroleum development problems. A. Bentz. *Erdöl u. Kohle*, 1951, **4** (5), 231–5.—Scientific exploration of the Northwest German oil region, dating from 1934, led to the discovery of a multiplicity of types of salt-dome fields, to the realization that structures like Nienhagen and Oberg are examples of older structural elements and not a consequence of salt-stock formation and that the latter process is not directly related to anticlinal axes, since it can take place in purely synclinal areas. In North Hanover and Schleswig-Holstein conditions are especially complicated, because there are salt beds in the Rotliegendes and Keuper as well as in the Zechstein. Evaluation of the full prospects of the salt-dome fields awaits solution of problems such as the chronology of the salt movement and the structure of the highly disturbed zone around the uplifts.

The most important discovery in Northwest Germany was the anticlinal fields in Emsland, for not only did this open up large production, but it was found that the anticlinal zone extended eastwards into South Oldenburg. Furthermore, it led to the discovery that inter-salt stock areas previously dismissed as simple synclines actually have complex tectonic histories with pre-Tertiary and pre-Cretaceous buried structures in which fields like Suderbruch, Eldingen, and Hohne were found. Palæogeographic research shows that the petroliferous Rhaetic, Lias, and Dogger sands of the buried structures do not extend over great distances but either become clayey or are replaced by sands at higher or lower levels. There is thus a good chance also of finding stratigraphic traps. Accumulation related to fault structures of the type so important in the Texas–Louisiana Gulf Coast so far have not been found, although there is extensive pre-Neocomian faulting as well as movements of a later date. Much more reflection seismic work is necessary.

In the German part of the Rhine graben so far only a few small fields have been found in marginal faulted blocks, the oil coming from various porous horizons ranging from Mid Keuper to Upper Jurassic. Reflection seismic surveys were commenced in 1950 to examine the almost unknown central part of the graben, and drilling has begun at Karlsruhe. Apart from fault accumulations, true stratigraphic traps are possible in the Oligocene and Miocene.

Work in the Molasse trough of South Germany is still in the preliminary stages. Drilling is undertaken primarily for stratigraphic and tectonic detail, and reflection seismic work has started. Earlier observations indicate that the area is petroliferous, but rapid discovery of economic accumulations cannot be expected. S. E. C.

21. German drilling activity in 1950. H. Lögters. *Erdöl u. Kohle*, 1951, **4** (5), 235–44.—In 1950 there were sixty-two test holes drilled in new areas, three of which were successful; sixty-four to explore new prospects in proven fields, twenty-four of which were successful; and 281 exploitation wells, 241 of which were successful.

A detailed account is given of the geological information obtained from these boreholes in the following regions: Schleswig-Holstein–Hamburg, East Friesland–Oldenburg–North Hanover, Emsland, the Wiehengebirgs foreland, South Hanover–Brunswick, Westphalia–Lower Rhineland. In the Rhine graben in South Germany a boring to reach the central part of the graben at Karlsruhe had reached a depth of 405 m in the Upper Oligocene by the end of the year. A large amount of structure drilling was carried out in the Molasse trough (Baden, Württemburg, and Bavaria), and in addition there were three deep tests which gave shows in the Molasse and Jura.

Reserves on 1.1.51 were as follows:—

	Proven, tons	Probable, tons	Total, tons
Emsland	18,900,000	6,950,000	25,850,000
Hanover	7,420,000	2,730,000	10,150,000
Hamburg–Holstein	1,120,000	840,000	1,960,000
Baden	45,000	20,000	65,000

Increases in the Emsland reserves are due largely to extensions in the Dalum and the Rühlertwist–Rühlermoor fields. In Hanover considerable increases were due particularly to discoveries at Eldingen and Suderbruch. S. E. C.

22. Geology of the Krosno district. J. Wdowiarz. *Nafta*, Sept. 1950, **6**, 249–51.—Aided by a geological map of the district the author presents a brief summary of lectures by himself and H. Swidzinski. M. S.

23. Arabian field found. Anon. *World Oil*, Sept. 1951, **133** (4), 334.—A well approx 13 miles south of Ras El Mishaab, offshore in the Persian Gulf, flowed medium-gravity oil on test. This is the ninth field to have been found in Saudi Arabia. C. A. F.

24. Aramco has two discoveries. Anon. *World Oil*, Aug. 1951, **133** (3), 282.—Daily output of Saudi Arabia now averages 740,000 brl, and two new fields have recently been found.

Safaniya 1, which was drilled 12,000 ft offshore, found oil in the Bahrein Zone, equivalent to the pay at Bahrein and Burgan; and Ithmaniya 1, approx 40 miles south of the Ain Dar field, found 30·5° oil at 6900 ft in pay believed to be in the Jurassic Arab, which is producing in the larger fields in Saudi Arabia. C. A. F.

25. Oil geology of Ethiopia. H. W. Straley. *Petroleum*, Sept. 1951, **14** (9), 241–3.—The geography and geology of Ethiopia is described. The grain of the structure of the country is controlled by the great Rift valleys, which cross the country from south to north. Mesozoic sedimentary rocks cover a crystalline basement and are, in turn, buried under Cenozoic lava. Oil and gas potentialities are divided into three catagories. The Eastern part of the Somali plateau and part of the Gulf of Aden are placed in the possible group, the western part of the plateau and the Afar desert are designated unfavourable and the rest of the country barren. R. E. P.

26. Report on the possibility of petroliferous beds in New Caledonia. R. Pomeyrol. *Rev. Inst. franç. Pétrole*, Aug. 1951, **6** (8), 271–82.—New Caledonia has many "serpentine" massifs, and about a third of the surface is formed of the more or less serpentinized peridotite. This rock has rich deposits of nickel, cobalt, and chromium. Its emplacement may be post-Eocene. In the northwest there is much metamorphic rock-gneiss, mica schists, and sericitic schists, supposedly Algonkian or Lower Palæozoic in age, although Jenser has suggested that they are Mesozoic. Permian or Triassic beds are the earliest of definitely fixed age. They outcrop extensively, and there is a littoral facies in the west in the Moindou, La Foa, and Boulouparis regions; a deep facies occurs in the Chaîne. The littoral facies has shales, conglomerates, and eruptive rocks (tuffs and rhyolites and andesites). The stratigraphic relationships between the littoral and deep facies are unknown.

Coals occur in a series which included marls, shales, sandstones, and conglomerates. Upper Cretaceous fossils are found in the upper part. There are brackish and marine forms. At times the coals are very bituminous.

The Eocene is several thousand metres thick, and includes conglomerates, sandstones, marls, clays, tuffs. Lower Eocene is seen in the Gouaro anticline. In the southwest there are Neogene rocks.

The sediments are extensively silicified.

The Permo-Trias could have yielded hydrocarbons, and the same is true of one zone of the Eocene, but the coal-bearing formations seem most favourable. Methane has been found in the Koné valley. The tectonics include numerous folds, and there are sandstones and limestones which could be reservoirs. Cap-rocks occur, but the intensity of some of the folding and the presence of serpentine are unfavourable features. The intrusions have broken and metamorphosed the rocks.

The Koumac Basin has Cretaceous which is extensively altered. The Eocene has limestones, schists, and breccias. There is intense folding and faulting. Heavy paraffinous oil seeps from the serpentine on the south of the Tuando massif, at about 100 m from steep Upper Eocene; it may be derived from the Cretaceous or Lower Eocene. A few shallow borings would be justified.

Geologically the Koné–Pouembat Basin is similar to the Koumac Basin, but the Eocene limestones are much less important, and they are lenticular. There are gaseous hydrocarbons at Poinda, probably from Cretaceous beds, but the seepage is only slight.

In the Poya–Bourail region is the Gouaro anticline. Eocene sediments rest on Permo-Trias or Cretaceous in the east. The southern flank of the structure is almost entirely under the sea. The length is 30 km. The eastern flank may be complex. No oil indications are known. A boring showed a good cap-rock overlying probable reservoir and source rocks. A really deep well seems warranted.

The southern Gulf area has Lower Eocene beds, but the full succession is in doubt.

The Anse Longue anticline runs parallel to the coast, and there is also the Montravel anticline. The Eocene limestones are bituminous at two points. There are fissure impregnations at both places. In 1907 a boring was made near the Noumea seep. An American army water boring at Montravel found a show in Upper Eocene beds dipping at 70° to the north. As a whole, this region does not seem very favourable.

Too much optimism is not warranted with regard to the oil prospects, because of the structure and the limited extent of favourable rock types. Nevertheless, there are chances of finding important oil accumulations. A 2000-m well should be drilled at Gouaro, with further work dependent on its findings. Several wells (up to 1000 m) should be drilled near the Koumac seep. G. D. H.

27. Supplementary data on the Tertiary formation of New Caledonia. N. Grekoff and Y. Gubler. *Rev. Inst. franç. Pétrole*, Aug. 1951, **6** (8), 283–93.—Studies have shown the presence of the Priabonian (Eocene) in two facies—paralic northeast of Nouméa, towards Tomo and in the region of Bourail–Gouaro, and reefal near Creek Aymes and in the Ouitoé peninsula. The Gouaro boring penetrated nearly 150 m of Priabonian, consisting of sandstones, sandy marls with sandy limestone intercalations, and coaly debris at the base. Bituminous organic matter was met at several levels. The beds show *Biphanispira*, *Pellatispira*, and *Discocyclinae*. Above the Gouara beds in the Nèra section is the Roche Percée, about 100 m thick, consisting of grey marls and sandstones, the latter having fragments of eruptives and metamorphics. Ascending the Nera series eventually gives breccias in the Bourail region, but in places there are signs of short reef phases. Tuffs and silicified limestones follow, and there is clear evidence of vulcanicity. At the top are " diabases," serpentine, and diabasic tuffs.

The reef facies is extraordinarily rich in algæ and large foraminifera. However, there is some terrigenous material which yields the same faunas as the paralic facies. The reef development is better at Ouitoé than at Creek Aymes. Some of the limestones have bituminous matter in fine fissures.

The succeeding beds belong to the Upper Aquitanian and Burdigalian. At Nepoui there is horizontal reef limestone alternating with marls. Typical Miocene reef limestone has been found 5 km southwest of Boulouparis, and this rests unconformably on Permo-Triassic. The limestone at first sight looks very much like the Eocene limestones of the Boulouparis region.

The known paralic formations are over 1000 m thick, and point to subsidence in a geosyncline bordering the reef zones. Orogenic movements must have occurred in the Oligocene.

The genus *Fabiania*, generally considered as of Eocene age, has been found to extend into the Miocene here. The Tertiary is similar to that of the petroliferous formations of East Borneo, New Guinea, and the northern Celebes. G. D. H.

Geophysics and Geochemical Prospecting

28. Evaluation of magnetics in the Delaware Basin. H. W. Hoylman. *World Oil*, Aug. 1951, **133** (3), 91.—The quality of many magnetic surveys made for the oil industry over the last twenty-five years is so low that they are of little value to the geologist. Certain surveys which have been made, such as that for the Delaware Basin in West Texas, can, however, be very useful, provided that the accuracy and reliability of the survey is determined, the anomalies are selected with care, and the predicted geological conditions are carefully checked with the known conditions.

The determination of the accuracy and reliability of airborne and ground surveys and the selection of anomalies are described with reference to the Delaware Basin survey. C. A. F.

29. The geophysical exploration of Emsland. H. Cloos. *Erdöl u. Kohle*, 1951, **4** (5), 250–9.—The last reflection seismic survey was commenced in autumn 1948, and the author gives a structural map on which, to cover the whole area, structure lines are drawn on reflecting horizons at the base of the Turonian, at the Jurassic–Lower Cretaceous junction, and in the Upper Trias. To supplement the discussion of this map a number of selected profiles is given.

The first reflection work was by the government survey in 1941, and this was followed by further work by various interests in 1942–45. The great importance of

Emsland was first demonstrated by these surveys, but it is unlikely the results would have been reached so quickly but for the earlier regional refraction survey made by the government in 1938. Prior to that there had been some magnetic and gravitational work, but the significance of anomalies then discovered could not be assessed, and the subsurface geology of the area was completely unknown. S. E. C.

30. Seismic velocities in Tertiary sediments, Upper Rhine graben. H. A. Rühmkorf. *Erdöl u. Kohle*, 1951, **4** (8), 469–70.—Reflection seismic surveys planned in areas covering the whole of the Upper Rhine valley from the Mainz Basin to the Swiss borders make verification of velocity relationships necessary for co-ordination of reflecting and geological horizons and prevention of errors in dip determination. Data was obtained from a borehole, Worms 1, from refraction work in Alsace, and from reflection shooting in an area to the west of the Forst field near Neudorf in Baden. It has been found that in Baden and in the Bienwald velocities in the Lower Tertiary, beneath the Niederröderner beds, are considerably higher than in North Germany. The increase is marked, and reaches 3500 to 4000 m/sec, these high velocities applying essentially to the Oligocene and Eocene. S. E. C.

31. Seismic refraction methods for defining the edges and flanks of salt stocks. G. Brinckmeier and H. v. Helms. *Erdöl u. Kohle*, 1951, **4** (6), 321–6.—Examples of various forms of time–distance curves are given for a number of salt stock structures at which there has been refraction shooting both from the foreland in the direction of the salt and vice versa. The importance of one or more marker horizons in the foreland for defining the edges and flanks of the salt is discussed. S. E. C.

32. Seismic prospecting at sea. J. Dienesch. *Bull. Ass. franç. Tech. Pétrole*, 31.7.51 (8), 12–19.—In the Gulf of Mexico for depths less than 50 m the instrument boat draws a cable with twelve geophones which rise on floats to 1·5 m above the sea-floor. Weights keep the cable down and straight. The first geophone is 105 m from the boat, and the last 300 m. Surface floats indicate the beginning and end of the line. A second boat handles the explosives. The charge is exploded at 150 to 1200 m to the side of the middle of the string of geophonee. It is suspended 1·5 m below the water surface by a float. Charges of 20 to 25 kg are used, and a radio signal marks the explosion. There is also radio communication between the two boats.

The position of the boats is fixed by Shoran via previously placed buoys. Including the Shoran boats, a total of six vessels are used.

The films have twenty-six traces : twelve not mixed, ten mixed, one time break, and three water breaks (direct arrivals through the water at about 1500 m/sec). Shots at two lateral distances for the same positions of the geophones are used for weathering data. The water surface is the plane of reference.

Off the coast of California the depth of water exceeded 300 m in places. The geophones were suspended from a float-supported cable at 5 m below the surface, with spring connexions to reduce shocks. Two lines were set at right-angles, each with twelve geophones. This permits dips to be obtained. The first geophone was 67 m from the recording boat, and the spacing was 19 m. Three boats handled the cables. The charge was suspended below the surface opposite the mid-points of the two lines. 40-kg shots were used. Shoran was used for fixing positions. Water breaks indicated the precise inter-relations of the geophones and shot point. Four boats only were used because operations were near to the coast. There was no appreciable weathering zone. No water correction was applied, and this caused no great distortion when the sea-floor had a gentle slope. Multiple reflections can be recognized if the depth of the water is known ; the first reflection is from the sea-floor, and this is compared with the direct water break.

70 to 150 shots can be made per day (ten to twenty times as many as on land).

G. D. H.

33. Significance of geophysics in the six-year plan of [Polish] petroleum industry. A. Kislow. *Nafta*, Oct.–Nov. 1950, **6**, 286–9.—Modern prospecting does not depend on luck but on scientific approach. The numerous methods at the disposal of modern geophysicist justify themselves by the discovery of many new fields in U.S.S.R. In Poland prospecting goes on in all likely areas. For the next five years large expenditure

on equipment is planned, but this is cheaper than wild-cat drilling. By intelligent co-operation with industry this investment may make the six-year plan much easier to achieve. M. S.

34. The interpretation of the results of mixed shooting. T. Krey. *Erdöl. u. Kohle*, 1951, **4** (7), 385–7.—The problem of two-fold mixing is analysed mathematically. It is concluded that in mixed shooting the reflections with small time differences must be evaluated with particular care, and a mathematical treatment of these enhanced requirements is developed. S. E. C.

35. High frequency capacity measurements in boreholes. H. Janssen and A. Schuster. *Erdöl u. Kohle*, 1951, **4** (4), 173–4.—For some years high-frequency measurements have been made in boreholes, in addition to porosity and resistivity, to obtain data on oil and water content. The method has worked well in the Reitbrook field, where the reservoir rock is chalk, and also in Emsland where the reservoir is in fine sand. Previous high-frequency arrangements used a coil coupled to the frequency-measuring circuit, and its capacity, self induction, and damping varied with the surrounding rocks. To increase the capacity effect and thereby improve the reaction to the presence of water, the coil was substituted by a rod antenna of length approximately 1% of the wavelength used. Results obtained with this set-up are discussed. The evaluation of results has not yet reached the stage where exact determinations of oil, water, and permeability can be made, but improvements are possible, and more sensitive apparatus is being built. S. E. C.

Drilling

36. West Texas preview of tomorrow's oil well rigs and drilling methods. R. Sneddon. *Petrol. Engr*, July 1951, **23** (7), B-60.—Interesting developments in the unitization of large drilling rigs and planning of drilling operations are described.

Unitization enables large rigs to be moved intact except for pumps and tanks. The derrick is jacked on to wagons and towed by tractors. The entire rig can then be moved in 6 to 7 hr instead of ten to twelve days as formerly.

Using two deep-drilling rigs and one completely unitized smaller rig, a new drilling procedure has been adopted.

The first 6000 or 7000 ft of hole are drilled with the small rig, and the hole then completed with one of the larger rigs. Considerable savings in initial investment and operating costs are obtained. C. G. W.

37. Drilling and production problems relating to the Worland, Wyoming, field. L. L. McDonald and J. I. Morris. *Petrol. Tech.*, May 1951, **3** (5), 14–17.—The Worland discovery was based on reflection seismograph work, and a well started in 1945 was taken to 11,379 ft in the Big Horn dolomite. It was plugged back to 10,131 ft and tested at 9990 to 10,055 ft through perforations opposite the Embar. Dangerous amounts of H_2S were obtained in a test, and the completion gave 165 b.d. of 43·5°API oil with a G.O.R. of 4900 to 5000. An abnormal production decline was found to be due to plugging of the well with hydrates (some 2800 ft). Corrosive gases attacked pressure gauges and tubing, etc., the gas escapes being dangerous in more ways than one.

Various muds were tried, caustic quebracho proving the most satisfactory and economical generally. Lost circulation is most serious in reducing the permeability of the producing horizon.

The oil gravity is lower in the upper part of the structure, and the G.O.R. is higher. Perforating was not very satisfactory, and casing is now usually set in the top of the Embar. Galvanized lines treated with a corrosion-preventing compound were found necessary for swabbing. Acidizing improved well productivity.

Hydrate formation was reduced most efficiently by a hot-oil-circulating system. A check valve was fitted to prevent escape of well fluids into the circulating system if failure occurred.

Individual tank batteries were installed at first to avoid unnecessary transport of dangerous gas. Escape of toxic gases was prevented, as was the entry of air. Persons

on tank walkways had to wear gas masks, while a second equipped party remained at the fire wall.

Breaks in tubing may have been partly due to thermal stresses and partly to hydrogen embrittlement.

Arrangements were made for the gas to be treated for the commercial recovery of sulphur from H_2S, the sweetened gas being returned for gasoline recovery. The conventional absorption and fractionation plant can process 30 M.M.c.f. of sweet Embar gas per day, recovering 70% of the propanes, 95% of the butanes, and all the pentanes plus. The crude-stabilization plant removes H_2S from the crude. The daily yield is 320 tons of sulphur, 10,000 gal of propane, 8500 gal of butane, and 12,000 gal of gasoline.

Now wells deliver oil into a gathering system operating at 325 p.s.i., which carries it to central separators. The field has been unitized.

Three Frontier completions have been made, and these too gave hydrate trouble.

G. D. H.

38. Construction of drilling equipment as a factor influencing the progress of drilling technique. S. Karlic. *Nafta*, Oct.–Nov. 1950, **6**, 295–307.—A review of drilling methods with special attention to efficiency. Author compares different motors, gear-boxes, fluid-drive mechanisms, and pumps for drilling, extraction of string and circulating mud. After making do with imported machinery for the most immediate needs of industry, Poland should go forward to own manufacture sponsored by the state. M. S.

39. Drilling equipment. M. F. Hazel. *Petrol. Engr*, Apr. 1951, **23** (4), B-16.—Developments in rotary drilling equipment during the past fifty years are briefly described. C. G. W.

40. Science and technique in Soviet Union (monthly review). Anon. *Nafta*, Oct.–Nov. 1950, **6**, 332–3.—Deals with: (1) high-speed drilling; (2) mechanism for manipulating the kelly; (3) foolproof valve for commencing gaslift. Drawings included. M. S.

41. The drilling technique in the Soviet Union as an example for the Polish oilfield practice. Anon. *Nafta*, Oct.–Nov. 1950, **6**, 278–83.—Tabulated data of Soviet drilling over the period 1920–41 shows that in 1920 it took eighteen months to drill a well, and in 1941 it took only ten weeks. That drilling per 1000 tons of crude rose from 1·56 m to 60·5 m, and electricity was the driving power in 60% of the cases as compared with only 5% in the U.S.A. Fuller analyses of data available shows to the Polish industry the method of lowering the cost of drilling. M. S.

42. Standardization and working standards in drilling practice. A. Waliduda. *Nafta*, Oct.–Nov. 1950, **6**, 307–13.—In the coming six years it will be necessary to standardize equipment for drilling and ancillary work to avoid waste. In an analysis of drilling procedure the author finds that approx two-thirds of the work is independent of nature, and so could be improved by application of time-and-motion study. M. S.

43. Well completion practices. T. A. Huber, T. O. Allen, and G. F. Abendroth. *Petrol. Engr*, Apr. 1951, **23** (3), B-36.—Developments in well-completion techniques are reviewed. These include the use of modified cements, gun and jet perforating, formation-fracturing and sand-screening techniques.

The importance of accurate depth measuring and measuring correlations is emphasized.

The lines along which completion techniques should develop are indicated.

References are appended. C. G. W.

44. Well completion practices in Oklahoma. K. M. Fagin. *Petrol. Engr*, Mar. 1951, **23** (3), B-54.—Dual completion in Oklahoma is of three types:—

(*a*) *Both formations producing through same flow string.* This method should only be used when bottom-hole pressures, drives, and gas–oil ratios are the same, or similar.

(*b*) *Normally separated completions.* The shallower formation produces through the

annulus the deeper through the tubing. The formation fluids are separated by a packer.

(c) *Cross-over completion.* The deeper horizon produces through the annulus and the shallower zones through the tubing. C. G. W.

45. Drilling and completion practices in Church Buttes fields. W. C. Dinger. *Petrol. Engr*, July 1951, **23** (7), B-16.—The structure is an elongated anticline, the production being from a Cretaceous sandstone at depths between 12,400 and 13,000 ft.

Drilling is straightforward, no problems of crooked hole, lost circulation, etc., being encountered. The only mud problem is when drilling a sodium carbonate formation. This, however, is treated without difficulty.

A surface string of 13¾-inch casing and an intermediate string of 6000 ft of 9⅝-inch casing are set, the latter being cemented with 600 brl of cement. While drilling out the cement plug the mud is converted to a calcium-base emulsion mud using oil, lime, and calcium liquosulphate.

The production string is 6⅝-inch previously tested to 6000 p.s.i. before being run. After cementing, the string is tested to 5400 p.s.i.

The tubing head is tested to 10,000 p.s.i. before installation.

Drilling time is 120 to 125 days using 136 to 146 bits. The producing zone is drilled with diamond-core heads using two or three diamond heads.

Initially wells were completed with a packer on the tubing, later wells have been completed without the packer.

Initial shut-in surface pressures are about 5400 p.s.i. with flowing pressures of 3000 to 4500 p.s.i. Flow-line pressures are 950 p.s.i., and heaters are necessary on flow lines to prevent hydrate formation.

Production is 6 to 8 brl 53 API condensate and 1 to 2 brl/million cu. ft. of gas with reservoir pressure of 7040 p.s.i. and temperature 250° F. The reservoir gas is a retrograde fluid, and no liquid phase is present in the reservoir. C. G. W.

46. Selection and evaluation of wells for hydrafrac treatment. J. W. Maly and T. E. Morton. *Petrol. Engr*, June 1951, **23** (6), B-28.—In the selection of wells for applying the hydrafrac treatment, the following factors should be considered :—

1. *Permeability.* Generally a greater increase in production is obtained in formations of low permeability (*i.e.*, 150 darcies) than in high-permeability zones.

2. *Reservoir energy.* With water and gas-cap drives the possibility of excessive break through of water or gas must be considered.

3. *Production history.* Most successful in cases where the initial rate of production declined rapidly to a low fairly constant level.

4. *Work-over history.* The results of prior shooting, or combined hydrafrac and acid treatment, vary considerably from well to well. Previous work-overs should give valuable information on the state of depletion of the well and the condition of the well bore.

5. *Formation characteristics.* Generally short sections of formation respond better than long sections. Hence with thick intervals it is best to perforate short intervals through casing or to segregate sections by means of packers.

Shale breaks vary in their effect on the treatment. Soft shales should be isolated from the fracturing fluid. Hard shales or shale lenses can generally be ignored.

Dirty or trashy sands generally react well to the treatment.

6. *Condition of well equipment.* All well equipment must withstand the applied pressure.

The important considerations are summarized and listed. C. G. W.

47. Power equipment revolutionizes drilling operations. *Petrol. Engr*, Aug. 1951, **23** (9), B-67.—A fully-automatic deep-drilling rig is described. C. G. W.

48. Large portable rig. K. M. Fagin. *Petrol. Engr*, Aug. 1951, **23** (9), B-14.—The rig is 131 ft bulge-type rated for 537,000 lb powered by three, six-cyclindered gas engines. The draw-works with built-in selective transmission, engines, and compounding transmissions are unitized on skids. Controls are pneumatic.

Parts of the rig are described and illustrated. C. G. W.

49. Ultra-modern diesel electric rig combines flexibility and portability. R. Sneddon. *Petrol. Engr*, May 1951, **23** (5), B-13.—Unusual features of this rig are : (*a*) operates on self-generated D.C. electricity ; (*b*) draw-works set at ground-level below derrick floor ; (*c*) is fully insulated and ventilated.

The rig will handle a hook load of 750,000 lb with ten lines strung. Use of pneumatic and electric controls gives finger-tip operation of the complete rig.

Amphidyne control of generator output gives a high degree of control, selectivity, and sensitivity on the electric motors.

Other features are described. C. G. W.

50. Radio-activity well logging anomalies. J. L. P. Campbell. *Petrol. Engr*, June 1951, **23** (6), B-7.—The correct interpretation of apparent anomalies on radioactivity well logs is of considerable importance.

Such anomalous results may be due to :—

1. *Deposition of radioactive crusts.* These crusts are associated with production of water, and are deposited opposite the zones of greatest permeability.

2. *Presence of lignite.* Lignite gives the only known radioactive value below the average shale line. The neutron log separates lignite from sand, whereas the gamma-ray log shows lignite as sand.

3. *Bentonite glauconite and organic marine shales.* Radioactivity intensity varies throughout shales. Dark shales and shales with a high potassium content give high gamma-ray values. The neutron curve gives the normal shales line.

4. *Casing.* Overlapping or parted casing, casing shoes, and fluid levels cause anomalies in radioactivity logs.

5. *Gas sands.* Studies of neutron and gamma-ray logs can be used to obtain an indication of the fluid content of a formation. It may eventually be possible to distinguish between gases, distillates, oils, or mixtures.

The instruments used in radioactivity well logging are briefly described and their applications discussed. C. G. W.

51. Underground magnetic logging device. Anon. *Petrol. Engr*, June 1951, **23** (6), B-87.—An instrument for magnetic logging of boreholes had been developed.

It is expected that magnetic logs will assist in the interpretation of surface and airborne magnetometer surveys and also in the determinations of subsurface structures. C. G. W.

52. Optimum use of coring electric logging and other testing methods in exploratory wells. Pt 2. J. E. Walstrom. *Petrol. Engr*, Mar. 1951, **23** (3), B-25.—Pt 2 of a comprehensive review of exploratory drilling practices. Testing and logging procedures and their applicability to various types of formation are classified, and drilling conditions outlined.

Recommended procedures are tabulated. C. G. W.

53. New developments in diamond coring. R. A. Watson. *Petrol. Engr*, June 1951, **23** (6), B-63.—The most recent development in diamond coring is a tool for drilling in soft steeply dipping shales. This consists of a full-hole-size diamond-core bit, inside which is placed a rock bit. The rock bit drills up the core as fast as it is drilled.

Diamond-core bits are proving valuable in whipstocking and side-tracking operations and also in drilling over " fish."

Some aspects of contract coring are discussed. C. G. W.

54. Carboxymethylcellulose in drilling mud. J. Stawitz. *Erdöl u. Kohle*, 1951, **4** (5), 272–7.—Of all the substances tested, the salts of carboxymethylcellulose proved to be the best protective colloids for drilling muds. They can be used with fresh mud or for regeneration of partly spoiled muds, and have been successful in drilling through sands and gravels, swelling clays and shales, chalk and anhydrite, and salt beds. The chemistry of carboxymethylcellulose is outlined. The outstanding characteristics of the material are largely due to the size, shape, and high electrical charge on the anion, and the effect of these factors on the properties of muds is discussed. It is normal practice in America to use pure carboxymethylcellulose, but in Germany the crude material is generally used. The commercial varieties differ in the length of chain,

degree of etherification, and content of the effective substance, and it had been found that for optimum effect and max economy it is best to select the type of product to suit the drilling conditions. Resistance to heat, pressure, moulds, and bacterial ferments is much greater than with other organic colloids, and preservatives are not necessary. S. E. C.

55. Importance of drilling mud for the improvement in rotary drilling practice. J. Wojcik. *Nafta*, Oct.–Nov. 1950, **6**, 290–5.—Describes the rôle and behaviour of drilling muds under different velocities. The importance of visc and density is pointed out. By addition of certain chemicals the action of muds on rocks may be varied. M. S.

56. Physical properties of gel cement. D. K. Smith. *Petrol. Engr*, Apr. 1951, **23** (4), B-7.—Bentonite has the following effects on oil-well cements: (1) increased slurry volume and lighter slurries due to higher water-cement ratios and better dispersion; (2) reduced hydrostatic pressure due to decreased slurry weight; (3) lower costs; (4) better perforation characteristics—lower compressive strength.

Field and laboratory studies have been carried out to determine the properties of gel cements.

Compressive strengths, thickening times, and slurry weights are tabulated for various percentages of bentonite with Portland cement and high-temp cement. C. G. W.

57. Exclusion of waters in wells. M. Ptak. *Nafta*, Sept. 1950, **6**, 251–9.—All water encountered in drilling must be excluded. In percussion drilling it is accomplished using mud or cement, in rotary drilling only cement can be used. The paper gives a summary of the methods and variations possible. A calculation is appended giving quantity of cement required for such an operation. M. S.

58. Methods of separation of drill cuttings from drilling fluid. J. Osiecki. *Nafta*, Aug. 1950, **6**, 220–4; Sept. 1950, **6**, 259–64.—Discusses theoretical background of the problem and practical methods of cleaning the mud. Due to the gelatinous nature of the mud it has to be subjected to considerable forces before it resumes its higher fluidity. Hydraulic and mechanical methods can be employed. Hydraulic are split into: (1) reservoirs; (2) graders; (3) troughs; and mechanical are (1) gravitational (with occasional shaking) and (2) centrifugal kinds respectively. Technical details of all five are given together with drawings and empirical formulæ. M. S.

59. Oil emulsion drilling fluids. H. W. Perkins. *Petrol. Engr*, Apr. 1951, **23** (4), B-60.—Available information on the properties and applications of oil-emulsion drilling fluids.

To prepare an oil-emulsion mud, a water-base mud, oil, and an emulsifying agent are necessary. The properties of the emulsion depend largely upon the emulsifying agent used. Most normal drilling fluids contain emulsifying agents, and the combined effect of these agents and the added agent should be considered.

Emulsion muds may be prepared by adding gas oil, diesel oil, or crude oil to any of the normal water-base drilling fluids (*e.g.*, sodium base, lime base, or silicate).

The performance of emulsion drilling fluids may be summarized as follows: (1) drilling rate increased; (2) bit life increased; (3) hole condition improved; (4) effect on pumps variable—replacements of rubber parts more frequent; (5) economy —insufficient data; (6) geological interpretation—sidewall sampling adversely affected, logging and coring unaffected; (7) completion—improved in some cases, never adversely affected. C. G. W.

60. Improvements in metallurgy and design reduce failures of drill string tools. F. Briggs. *Petrol. Engr*, May 1951, **25** (5), B-7.—Examples of types of failures occurring in tubular goods are presented.

Metallurgical and design methods which may eliminate many failures are outlined. C. G. W.

61. Collar-type elevator in deep casing operations. W. A. Wilson. *Petrol. Engr*, May 1951, **23** (5), B-76.—Laboratory tests in which casing and coupling sections were tested to failure point with slip-type and collar-type elevators have been carried out.

Within the limits of accuracy of the equipment used, there is no evidence that joint strength is reduced with collar-type elevators. C. G. W.

62. Straight hole drilling in crooked-hole country. G. C. MacDonald and A. Lubinski. *Petrol. Engr*, May 1951, **23** (5), B-54.—Any techniques and equipment proposed for the drilling of straight holes or the correction of crooked holes, must be assured of mechanical and economic success.

Straight-hole drilling may be accomplished as follows :

(*a*) Use of oversized drill collars, *i.e.*, a collar which cannot be washed over in the hole being drilled. The principal hazard with oversize drill collars is sticking due to settling of cavings on cuttings. High circulation rates are necessary with oversize drill collars.

(*b*) Use of bit weight below critical values or the use of stabilizers at the points where buckling occurs. A previous paper on drill string buckling (see Abs. No. 1761, 1951) established the weights on the bit and the point in the string, at which buckling occurred. It is proposed that weights less than the critical should be carried on the bit, or that where such weights give insufficient penetration, stabilizers should be placed at the points where buckling is anticipated.

(*c*) Rotary percussion drilling. Although not yet a fully developed drilling method, the hammer drill enables drilling to be carried on with comparatively small weights on the bit.

(*d*) Combination diamond core barrel and rock bit. The rock bit is placed inside and above the core bit and grinds the core as fast as it is cut.

(*e*) Rat-holing ahead and reaming the pilot hole. C. G. W.

63. New borehole inclinometers. H. Richter. *Erdöl u. Kohle*, 1951, **4** (1), 18–22.—In recent years three new, non-directional inclinometers for borehole surveys have been developed : the Gräfe NO 5, the Multiplex and the Becklot. The Gräfe is operated by clockwork and records the position of the point of pendulum after a given time interval. The Multiplex gives a similar record, but is operated by a hydraulic mechanism (see Abs. No. 50, 1950). In the Becklot instrument the record is made by a small hole being punched in the paper which is attached to the pendulum. Details of the construction of these instruments are given. A short account is also given of the improved version of the Fotolot 48, a directional inclinometer of a type similar to the Eastman. S. E. C.

64. Guy rope anchoring for drilling masts and derricks. O. Stötzner. *Erdöl u. Kohle*, 1951, **4** (6), 340–3.—Specification DIN 4111 requires guy-rope anchors to have a satisfactory safety factor, but gives no statement of what this should be or of methods of calculation. On the basis of a mathematical analysis author deems a safety factor of 1·5 sufficient. A numerical example illustrating the calculations and their application is given. S. E. C.

65. How 2-way radio aids in drilling. P. A. Greenmeyer. *Petrol. Engr*, June 1951, **23** (6), B-40.—Radio systems of the single-frequency type giving communication between cars, stations, and rigs are claimed to save time and improve efficiency during normal operations. At times of emergency the time saved in getting equipment or personnel to the site needed may be vital.

Distances over which the system has been used are as follows, car to station—50 miles, car to car 20 miles, rigs to base station 90 miles. C. G. W.

66. Weather radar and offshore operations. W. W. Hildreth, Jr. *Petrol. Engr*, July 1951, **23** (7), B-7.—The use of radar in improving the accuracy of weather forecasting, particularly squalls and hurricane conditions, is discussed with particular reference to offshore operations. C. G. W.

67. Submersible 1800 h.p. drilling barge operates in Corpus Christi Bay. G. O. Danielson. *Petrol. Engr*, June 1951, **23** (6), B-15.—The rig is an 800,000-lb 136-ft galvanized-

steel derrick (hook-load capacity 430,000 lb) capable of drilling 15,000 ft, mounted on a submersible hull 11 × 45 × 211 ft.

The rig is powered by three 600-h.p. diesels and is drilling 8000-ft wells with 5-inch drillpipe and jet bits in about twenty days from spudding to setting casing.

Cementing and logging equipment is transported to the rig by barges. C. G. W.

Production

68. Further developments in the laboratory determination of relative permeability. B. H. Caudle, R. L. Slobod, and E. R. Brownscombe. *Petrol. Tech.*, May 1951, **3** (5), *AIMME Tech. Paper No.* 3056, 145–50.—The experimental techniques for the solution-gas and dynamic-displacement methods of obtaining relative permeability data are described. There is also a description of a routine procedure for obtaining two-phase relative permeability data for small core samples.

It was found that decrease of the flow rate gave a lower relative permeability to oil at a given oil saturation for the gas–oil dynamic flow system. When the initial water content before starting the measurements was varied it was found that the oil and water relative permeabilities obtained subsequently differed for a given saturation. It is considered that except for special studies the amount of water present at the start should be the irreducible minimum saturation.

Some results of three-phase flow are given. G. D. H.

69. Vaporization characteristics of carbon dioxide in a natural gas–crude oil system. F. H. Poettmann. *Petrol. Tech.*, May 1951, **3** (5), *AIMME Tech. Paper No.* 5058, 141–4.—Equilibrium vaporization ratios have been determined for carbon dioxide in a natural-gas–crude-oil system for temperatures of 38°, 120°, and 202° F and pressures ranging 600 to 8500 p.s.i. No significant variation was shown in the equilibrium vaporization ratios of the hydrocarbons or the carbon dioxide itself for carbon dioxide concentrations up to 12 mole-% in the composites. The carbon dioxide was found to be more soluble in crude than in distillate.

The ratios, together with those determined for carbon dioxide in a distillate and various binary mixtures of hydrocarbons, should provide a fairly complete picture of the vaporization characteristics of carbon dioxide in hydrocarbon mixtures.

The experimental technique is described, and the experimental results are presented in tabular and graphical form. G. D. H.

70. De-emulsification of crudes. S. Rachfal. *Nafta*, Sept. 1950, **6**, 264–8.—Author describes briefly early work on this problem when heating was the only means of de-emulsification. Modern methods fall into three categories: mechanical, electrical, and chemical. An example of each is given, with special attention to the last method. The paper concludes by describing a method invented by the author. M. S.

71. Mineralogical studies of California oil-bearing formations. I. Identification of clays. P. G. Nahin, W. C. Merrill, A. Grenall, and R. S. Crog. *Petrol. Tech.*, May 1951, **3** (5), *AIMME Tech. Paper No.* 3059, 151–8.—The initial phase of a study of the role of clay in reservoir performance is reported. The silt and clay content of the reservoir rocks have the highest surface per gram, and are the most chemically reactive of the reservoir rocks. The analytical techniques employed include X-ray diffraction, electron microscopy, thin-section petrography, infra-red spectroscopy, and base exchange. A suite of the more important clay minerals was examined by these techniques to obtain data for comparison with core material. No single method appears to be fully diagnostic for all the clay minerals, but the infra-red technique shows considerable promise in this direction.

The fine material in cores was separated by a method which combined elutriation and continuous centrifuging, while fractionation was subsequently done by centrifuging.

In examining cores from the Stevens zone and Gatchell sand it was concluded that the former had a predominance of montmorillonite or illite, while the latter had kaolinite or anauxite. These findings agree with the observations that the Stevens zone is sensitive to fresh water, while the Gatchell sand is not susceptible to damage by fresh water. G. D. H.

72. Continuous electronic recording of the water content of oilfield emulsions. P. O. Engelder. *Petrol. Engr*, Apr. 1951, **23** (4), B-28.—An electrical method for the measurement of water content of emulsions is described. The method depends on the polarity of the disperse phase (*i.e.*, water), and consists of measuring the dielectric constant and loss tangent of the fluid stream. The loss tangent is defined as a function of the angle by which the current (A.C.) through a dielectric fails to lead the voltage by 90°. Thus the loss tangent is a function of power loss in a dielectric.

The two properties are measured by passing the fluid stream through a capacitor consisting of two concentric cyls. Radio-frequency power is fed to the capacitor, and charges in dielectric constant loss tangent are recorded as varying voltage at the input of a vacuum-take voltmeter.

Recoverable linearity of results is obtained when operating frequency, electrode geometry, and circuit constants are suitably chosen.

Accuracy is about $\pm 0{\cdot}3\%$ under best operating conditions. Care should be taken to calibrate the apparatus using the same crude oil as that in the natural fluid stream. This is necessary, since changes in aromatic or other polar hydrocarbons will cause anomalous readings. C. G. W.

73. Sand and gas control in pumping wells. N. C. Wells. *Petrol. Engr*, May 1951, **23** (5), B-45.—The application of pump-powered filters to wells with severe sand trouble is discussed.

The pump-powered filter consists of a moving pack of glass agates, the movement being effected by the well fluid. The agates are generally of 0·30 inch dia.

Certain types of gas trouble can also be corrected by the use of these filters.

It is emphasized that pump-powered filters are not applicable to all cases of sand and gas trouble and that care is necessary in installation if the method is to be effective. C. G. W.

74. Problem of band-wheel pumping unit dynamics. Z. Turkowski. *Nafta*, Aug. 1950, **6**, 225–9.—Author in his researches for Polish Petroleum Institute has been confronted with cases of repeated breakdowns of band-wheel units in use in many oilfields. By means of graphical analysis of a hypothetical system he produces a scheme of loading and also suggests that greater efficiency can be attained by planning a time-table of operations for wells which only pump a few hours a day. M. S.

75. Design of oil well pumps. M. C. Turner. *Petrol. Engr*, Aug. 1951, **23** (9), B-34.—The selection is discussed of plunger-type pumps to overcome such pumping difficulties as: (1) entrained sand; (2) low API gravity crudes; (3) scale deposition; (4) entrained gas; (5) corrosion. C. G. W.

76. The plunger lift. B. Schweiger. *Erdöl u. Kohle*, 1951, **4** (8), 491–3.—The first experiments with the plunger lift were made by the author in Boryslaw in 1917, but the apparatus was developed independently by the Hughes Tool Co. in Texas in 1927. The first wells to be fitted with the lift in Galicia started work in 1939, and ran continuously for seven months until war commenced. The total cost per ton production was one-half to one-third that of other pumping methods. The operation of the plunger lift is described. S. E. C.

77. Effect of free gas saturation on oil recovery by water flooding. C. R. Holmgren and R. A. Morse. *Petrol. Tech.*, May 1951, **3** (5), *AIMME Tech. Paper No.* 3055, 135–40.—It has been reported that the presence of free gas has led to lower residual oil saturations in sands undergoing water flooding than would otherwise arise. The degree of improvement depends on the conditions. If the gas occurs inside the oil, then since there seems no reason why the non-wetting phase saturation (oil plus gas) should differ in the two cases, which automatically means a lower oil saturation in the presence of the free gas. In practice, the presence of gas in the oil will reduce the permeability of the formation to oil, thereby causing water break-through at a lower water saturation.

In previous experimental work the effects of an initial gas saturation were uncertain, because the saturation may have varied during the flooding process. The present work used small pressure differentials relative to the absolute pressure, so that there

was little variation in gas saturation. A 5-ft long 5-inch diameter sandstone core was used. Its average porosity was 27·4% and average permeability 612 mD. Piezometer and conductivity rings were fitted. The core could be used at pressures up to 1000 p.s.i. and temperatures up to 150° F. The oil was methane-saturated at 388 to 517 p.s.i., its viscosity then being 0·75 to 0·85 cp. The brine had 33,000 p.p.m. of NaCl, with sodium nitrate as a corrosion inhibitor.

The brine-saturated core was oil-flooded until the oil saturation was 73·6 to 70%. The oil was methane-saturated. Gas drive then went on with later oil addition, until the required residual gas saturation was reached. The core was then ready for water-flooding. Other procedures were also used to get the necessary free gas.

The maximum pressure gradient in flooding was 2·7 p.s.i/ft, and flooding continued to a water/oil ratio of over 100. At low gas saturations the reduction in oil saturation below that of the reference run was only slightly less than the free gas saturation. At higher gas saturations there was a diminution in the amount of additional oil recovery, and at 25·3% gas saturation the total reduction was 15% of the pore volume. This accords with decreased relative permeability to oil.

It was found that a given gas saturation established by the gas-expansion technique yielded a smaller increase in oil recovery than a gas saturation created by the flooding at constant pressure technique.

As a result of the above experiments and the supplementary investigations it was concluded that: (1) the free gas in water-flooding changes oil-relative permeability and occupies space which would otherwise be filled with residual oil; (2) the reduction in residual oil can be computed from the appropriate relative-permeability characteristics. The possibilities must be considered relative to the reservoir-rock properties. Less water would be needed for fill-up, and flooding could be started earlier, while there would be a more uniform production rate. G. D. H.

78. Additional oil production through flooding with carbonated water. J. W. Martin. *Petrol. Engr*, Aug. 1951, **23** (9), B-58.—The injection of dilute solutions of carbon dioxide has been shown to effect a considerable increase in oil recovery. The process is believed to be in effect a chemical treatment of the reservoir. Influential factors are: (1) degree of carbonation; (2) temperature of oil sand; (3) pressure of the oil sand; (4) nature of crude oil; (5) natural-gas content.

The results of use of carbonated water are as follows: (1) increased injectivity; (2) increased production of a rich natural gas; (3) production of unsaturated hydrocarbons; (4) production of organic acids; (5) little or no corrosion; (6) no tendency to plug well; (7) carbon dioxide is largely recoverable. C. G. W.

79. Gas lifting by continuous flow. C. D. Fletcher and M. M. Livingstone. *Petrol. Engr*, Aug. 1951, **23** (9), B-42.—Modern gas-lifting practices for continuous flow demand sensitive and accurate control from the surface of a properly designed valve string.

The construction of a suitable valve, its mode of operation, and the location of valves in a valve string are described. C. G. W.

80. The Lacq reservoir rock. R. Gayral. *Bull. Ass. franç. Tech. Pétrole*, 31.7.51 (88), 20–9.—The Lacq reservoir rock is a Campanian limestone. The porosity is 25% in Lacq 1. 500 m away Lacq 2 showed dolomitized limestone with a lower porosity and impregnation, but it was fissured, and the output was far higher than in Lacq 1. Lacq 2, 4, and 7 show fissured dolomotized limestone; all other wells show less dolomite and a more uniform porosity.

Cores are taken to the laboratory, and as soon as possible the oil and water contents are measured by the use of toluene. The rock porosity is determined. The oil is undersaturated with gas, and this gas is lost together with some oil and water in bringing the core up the well. This loss is most serious in high-permeability cores. There is also some evaporation loss, while mud contamination occurs. Air permeabilities are measured on extracted core samples. Hydratable materials are lacking. The dolomite and calcite contents are determined.

The data are presented graphically against position in the well. In Lacq 3 porosities ranged 10 to 25% (mean 19·6%) and calcite 82 to 84%, dolomite up to 5%. Lacq 1 had 30% of connate water with permeability of 10 to 15 mD in the first three cores. The last core had 90% water with 2 mD and 69% water with 15 mD in two samples. Lacq 4 produces from a fissured zone. The solid rock has very low permeability.

Electric logging does not give the fluid content of the Lacq reservoir, being more dependent on lithological variations than fluid saturation. Mechanical coring alone provides the required data.

Capillary pressure and restored state studies are being made. G. D. H.

81. Formation water and oil recovery. W. Rühl. *Erdöl u. Kohle*, 1951, **4** (8), 470–8.—Much work has been done on the water content of reservoir rocks, but most of it is devoted to theory and laboratory experiment, and water as a factor in production is given a subordinate place. The author therefore deals with the influence of water as seen in examples taken from the German fields of Wietze, Thören, Rickelshof (Heide), and Wesendorf. Porosity, permeability, grain size, pore shape, oil saturation, visc, and the form of energy can cause extraordinary variations in flowing and flooding conditions, and the examples chosen show that water is a driving force which can have both favourable and unfavourable effects on oil recovery. There are many stages between the two extremes exemplified by the Wietze field with its lost energy and recovery of only 14% and the Wesendorf field which has been systematically exploited and has the max recovery of 60%. The other two fields described are examples of these intermediate conditions; the Thören field is already appreciably flooded and has 22% recovery, while the Richelshof field, in which development of the limestone reservoir started under ideal conditions but, because of wartime needs, had its production rate forced up, has 34% recovery. S. E. C.

82. Development of spangseal tubing joint. J. L. Holmquist. *Petrol. Engr*, July 1951, **23** (7), B-54.—In order to overcome the leakage occurring with API tubing the following type was adopted.

The joint is of integral type with API threads. Leakage is prevented by a conical sealing surface on the pin and in the corresponding box section.

The joint is designed for nominal make-up of two turns beyond hand-tight. The seal interference is such that contact is made with the joint hand-tight. After make-up is complete permanent deformation of the pin occurs, and consequently a seal can only be obtained on repeated make-up by making-up further than the recommended two turns.

Consequently the joint is recommended for flow strings only. C. G. W.

83. Fracture contact affects energy behaviour between wells. F. R. Cozzens. *Petrol. Engr*, July 1951, **23** (7), B-35.—In some areas producing wells or newly drilled wells may of necessity be located close to abandoned wells.

The efficiency of primary or secondary recovery will be considerably impaired if channelling occurs between the drainage radius of the abandoned well and that of the new well.

Rather than distort the well-spacing pattern excessively, it may be desirable to retain a shell of unfractured sand between new wells and abandoned wells. Consequently the effective radius of the new well must be controlled. Such control is usually effected by :—

1. *Heating.* Applied particularly in wells 50 to 75 ft from a known abandonment. Heat source may be electrical, steam, air–gas mixtures, or by chemical action. The well bore is maintained at 200° to 230° F for about 48 hr. The well is bailed, washed down, and the procedure repeated a few days later.

2. *Low-strength explosives.* For restricted fracturings radius-control normal practices are modified. Nitroglycerin dynamite of 40% strength or its equivalent in gelatin or solid nitroglycerin is fired in a substantially dry hole. If possible the explosive is run in two shells of small diameter (2 to 2½ inches) run side by side.

Shot concentration is 2 to 4 quarts/ft using no stemming.

3. *Acid treatment.* 15 to 20% acids are used, the acid being injected down tubing by oil pressure (annulus being packed off).

Acid is not left in the formation more than 3 hr after the pump is disconnected. After the solution is spent, tubing and packer are withdrawn and the solution bailed from the well.

Further treatments may be applied if necessary. C. G. W.

84. Soluble plug systems used to control paraffin deposition. K. M. Fagin. *Petrol. Engr*, June 1951, **23** (6), B-54.—To control paraffin deposition in pumping wells the following system has been adopted.

A string of 1-inch tubing is run in the well parallel with the main tubing string. At a point below the normal occurrence of paraffin deposits the 1-inch string is coupled to the main string by a cross-over tee.

Production is taken through the 1-inch string. Periodically a soluble plug is pumped down the 1-inch string, production being taken through the main string. When the accumulated paraffin is cleared, the fluid direction is reversed and the plug pumped back through the 1-inch string. C. G. W.

85. Subsurface pressure measurements. E. Nebe. *Erdöl u. Kohle*, 1951, **4** (4), 169–73.—Static-pressure measurements give information on relative permeability, production index, pressure distribution, comparison of wells, productivity, reserves, and the rationalization of field development. In comparison with static pressures, flowing pressures are much less important. They provide data on friction loss in tubing, the G.O.R. and saturation pressures. The flowing pressure should be measured at the bottom of the well to minimize irregularities due to gas expansion in the upper part of the tubing. S. E. C.

86. A method of estimating gas reserves. R. E. Davis. *Oil Gas J.*, 27.9.51, **50** (21), 99.—The three basic methods of estimating gas reserves are briefly described; the analogy, production/performance, and volumetric methods.

Proved reserves of the major gas-producing areas of the U.S.A. have been calc by the volumetric method, in which the reserve is computed from the amount of pore space present in the reservoir, the vol of gas in the pore space, and a recovery factor based on production experience.

A map of the important sedimentary basins and the major gas-producing areas of the U.S.A. is included. C. A. F.

87. Helium tracer gas studies. E. M. Frost, Jr. *Petrol. Engr*, Mar. 1951, **23** (3), B-7.—A field study of the use of helium as a tracer in gas-injection projects is described.

With the project chosen, natural gas was being injected through five wells, at a total rate of 27 M.c.f./day. One of these wells was chosen as a helium-tracer injection well. Natural gas was being injected into this well at a rate of 3 M.c.f./day at a wellhead pressure of 1270 p.s.i. The natural gas in use had a helium concentration of 0·022%, and this was increased to 1% for a period of five days. To achieve this, helium was throttled from cyls through an orifice meter into the suction of the gas compressors.

Samples of gas were taken from producing wells and helium content plotted against time for each well. A cumulative plot of helium content against time indicates the change from displaced-function gas to injection gas, as the end of economic cycling approaches.

From the results of the project the volumes of the flow channels between injection and producing wells have been calculated.

The results indicate that most of the gas flows along one narrow channel. Approximately 95% of the original volume of tracer was recovered during the 1½ years subsequent to the injection of the traces. C. G. W.

88. Stationary compressors for oilfield use. W. Müther. *Erdöl u. Kohle*, 1951, **4** (6), 236–9.—Details are given of the DEMAG range of gas compressors which have built-in, four-stroke gas-engine drives. S. E. C.

Oilfield Development

89. World production drops. T. A. Armstrong. *Oil Gas J.*, 27.9.51, **50** (21), 59.—World production in July 1951 averaged 11,703,800 b.d., compared with 12,003,900 b.d. in the previous month.

As a result of the cessation of Persian production there was increased production in Kuwait (by 22·2%) and Saudi Arabia (by 8·1%). Canadian peak production of 157,500 b.d. was reached during the month. C. A. F.

90. Exploration at all-time record. Anon. *World Oil*, 1.7.51, **133** (1), 88.—902 exploratory wells were completed in the U.S.A. in May 1951, of which 168 (or 18·6%) were producers.

Tables show the results of exploratory drilling in the U.S.A. in May and the first five months, 1951–50, by districts, and a summary of the results of exploratory drilling. C. A. F.

91. Exploration 17·8% above 1950 period. Anon. *World Oil*, Aug. 1951, **133** (3), 106.—4793 exploratory wells were completed in the U.S.A. in the first six months of 1951, 854 more than in the corresponding period of 1950. Of these 932 found commercial production and 3861 were dry.

Tables show the results of exploratory drilling in the U.S.A. in June 1951, and first six months, 1951–50, by districts, and a summary of the results of exploratory drilling. C. A. F.

92. Completions rise 4·1% over 1950 period. Anon. *World Oil*, Aug. 1951, **133** (3), 77.—21,239 wells were completed in the U.S.A. in the first six months of the year, 844 more than in the comparable period of 1950.

A table gives well completions in the U.S.A. in June 1951, and first six months 1951, by districts. C. A. F.

93. Completions continue at high rate in July. Anon. *World Oil*, Sept. 1951, **133** (4), 84.—3915 wells were completed in the U.S.A. in July 1951, as compared with 4125 in the previous month. Cumulative total for the year at the end of July was 25,105, 2·9% higher than in the comparable period of 1950.

A table gives well completions in the U.S.A. in July 1951, and first five months, 1951, by states and districts. C. A. F.

94. U.S. may produce 8 million barrels daily by 1955. R. E. Spann. *World Oil*, Sept. 1951, **133** (4), 111.—A summary of forecasts of U.S.A. annual productive capacity for 1951 to 1955 is given. This includes the results of a " productivity trend " analysis made by the U.S. Petroleum Administration for Defence which indicates that crude production in the U.S.A. can be increased from approx 6·8 million b.d. in 1951 to approx 8 million b.d. in 1955, assuming approx 43,400 wells will be completed annually during the next five years. C. A. F.

95. Completions 2·3% above 1950. Anon. *World Oil*, 1.7.51, **133** (1), 50.—3673 wells were completed in the U.S.A. in May 1951, 208 more than in the previous month. Total for the first five months of 1951 was 17,029% higher than for the same period of 1950.

A table gives well completions in the U.S.A. during May, and first five months of 1951. C. A. F.

96. California's San Ardo field again active. D. H. Stormont. *Oil Gas J.*, 20.9.51, **50** (20), 260.—Productive area of the San Ardo field in the Salinas Valley, Monterey County, which was discovered in 1947, is approx 5500 acres, and approx 200 wells additional to the present 150 producers will be required to completely develop the field on 10-acre spacing.

The structure of the main part of the field is a broad anticline, and there are two productive sands at approx 2000 ft, the Upper Miocene Lombardia and Aurignac.

Drilling and completion practice in the field are briefly described. C. A. F.

97. Denver–Julesburg Basin scene of active drilling with 36 rigs operating. J. A. Kornfeld. *Oil Gas J.*, 20.9.51, **50** (20), 247.—Thirty-eight rigs are operating in the Denver–Julesburg Basin, which extends through southwestern Nebraska, northeastern Colorado, and southeastern Wyoming. Total daily production (restricted) is 9320 brl from twenty-five fields. Completion of a pipeline through the basin will enable production to be increased by approx 15,000 b.d.

Pays are Upper Cretaceous Dakota–Graneros sands, and productive depths varies from 3200 to 8700 ft.

Drilling and completion practice for the six fields are described. C. A. F.

98. Successful water flood. J. A. Kornfeld. *Oil Gas J.*, 11.10.51, **50** (23), 78.—As a result of water flooding in the Benton field, Franklin County, Oklahoma, during

the last twenty-two months, field production, which had fallen from 33,000 to 730 b.d. over eight years, has risen to 7500 b.d. and may reach 12,000 b.d.

The pay is Tar Springs sand at approx 2200 ft, which lies 1400 ft below an operating coal-mine; unusual well spacings and drilling methods have been required.

C. A. F.

99. German oil production. G. Schlicht. *Erdöl u. Kohle*, 1951, **4** (5), 260–72.—German production fell from a peak of 1·06 million tons in 1940 to 550,000 tons in 1945, but since 1947 it has risen steadily and reached 1·118 million tons in 1950. Neglect of exploration as well as over-production from existing fields were the reasons for the wartime decline, and there was also a fall in technical standards. The currency reform of 1948 and the possibility of getting the latest American equipment which was afforded by the Marshall Plan have been of the greatest importance in the expansion of the industry. The important advances and present state of drilling and production practice are outlined, and the economic position of the industry is discussed. A comparison of German conditions with American reservoir conditions is not valid if scale is omitted from consideration, and for this reason German oil will always be at a disadvantage compared with the great production from the classic fields. To complete this survey of the industry factors affecting personnel are discussed. S. E. C.

100. Primary aims of the six year plan of the [Polish] petroleum industry. Borecki. *Nafta*, Oct.–Nov. 1950, **6**, 274–8.—Amongst these is preparation of new bitumen (crude petroleum) bearing deposits in known or likely districts. This may involve further prospecting. Planned exploitation will help to make most of those which are producing. Intensive action against waste in refining also stands high on the list.

M. S.

101. Report sheds light on Russian output. Anon. *World Oil*, Aug. 1951, **133** (3), 274.—Russian crude output in 1950 is indicated to be approx 273,465,000 brl or an average of 749,200 b.d., of which Asiatic Russia produced 44%. Operations in the Maikop and Gronzy areas are reported to have been fully restored during the year, and developments of new areas in Asiatic Russia are reported. These include the Baskir, Kuibyshev, Turkman, Uzbek, Kazkh, and Tatar areas.

Eastern European countries are believed to have produced a total of approx 44,588,000 brl in 1950. C. A. F.

Transport and Storage

102. Oil transport by road tanker. Anon. *Petroleum*, Nov. 1951, **14** (11), 293–6.—Reviews recent developments in British-made road tankers. The present speeding up of handling techniques is noted, and a number of popular models of petroleum road-transport units are described. R. E. P.

103. Oilfield transport and lifting equipment. Anon. *Petroleum*, Nov. 1951, **14** (11), 217–300.—Reviews the wide variety of specialized transport equipment (mobile cranes, fork trucks, lift trucks, etc.) used in oilfields, pipeline operations, and refineries which are now available from British manufacturers.

One important development is the use of the Pagefield system, whereby a lorry is able to exchange a number of bodies whilst avoiding the necessity of having a large number of motive-power units. Economy in man-power is said to be considerable.

R. E. P.

104. Pipeline transportation, storage and shipment of liquefied petroleum gas. D. A. Roach. *Oil Gas J.*, 8.11.51, **50** (27), 286.—Storage equipment, a major expenditure in pipeline movement of LPG for butane and propane is discussed, together with volume measurement, product test, shipment methods using buffer slugs, and loading equipment.

The pipeline hydraulics of LPG are illustrated by tables showing differential heads developed by a centrifugal pump at constant speed, and line-pressure drop for various products. G. A. C.

105. Structural design of flexible protective casings for pipelines. M. G. Spangler. *Oil Gas J.*, 8.11.51, **50** (27), 193.—Results are given of extensive research which has been carried out at Iowa State College on the structural performance of flexible types of underground conduits, and its application to design of protective casings at railroad crossings presented. The properties of soil relative to buried circular pipelines are discussed; and a typical example of an underground pipe under a railway crossing illustrates a design procedure for this type of structure.

Ten figures and one table illustrate the article, and thirteen literature references are given. G. A. C.

106. Construction of a military portable and convertible gasoline storage tank. C. Meyer. *Bull. Ass. franç. Tech. Pétrole*, 1951 (89), 3–40.—Portable storage tanks were widely used by the U.S. Army in the second world war. These were based on type of tank in normal civilian demand in the U.S.A. Such demand does not exist in France, hence a simpler and cheaper design of tank was required for the French Army; necessity is stressed of using only components that can easily be supplied by French industry, and which can be manufactured by simple standard engineering operations. Plane instead of cylindrical walls are used, and main groups of tank components are reduced to three, framework, panels (side, bottom, roof), and sealing strips, the latter being of synthetic rubber. Basic design is a ten-sided tank of capacity 80 m^3 and weighing 36 kg/m^3; this can easily be expanded to a fifteen-sided vessel of 185 m^3 (25 kg/m^3). Constructional material is mild steel (40 kg/mm^2). Detailed illustrated technical description of tank design, components, assembly, stress calculations, etc., is given. V. B.

107. Collapsible storage tanks. Anon. *Petroleum*, Aug. 1951, **14** (8), 217–18.—The need for easily movable tanks for storing petroleum has led to the development of a 10,000-gal collapsible tank as a result of wartime trials and post-war research at Fort Belvoir, Virginia. The "pillow" container is made of Bura N synthetic rubber reinforced with inner and outer layers of nylon cloth. The inner layer is coated with nylon to serve as a barrier to prevent porosity, and a layer of Buna N is in contact with the gasoline. Results of a number of tests made on the fabric and the container are given. These show that: (1) 0·021 oz of gasoline pass through a square foot of fabric in 24 hr at 75° F using 91-octane gasoline; (2) one year's storage did not damage container; (3) it can be used at −45° F; (4) 12,000 gal water could be held safely. R. E. P.

108. Automatic gauging, sampling and testing. F. H. Warren. *Oil Gas J.*, 8.11.51, **50** (27), 271.—Recent developments in equipment are reviewed, particularly in regard to automatic tank gauging and temp-reading devices. It is suggested that use of automatic methods should be encouraged by inclusion in Code 25 of a provision permitting their use. G. A. C.

109. Supports for vertical pressure vessels. Pt III. F. E. Wolosewick. *Refiner*, 1951, **30** (10), 143.—The stresses set up in ring stiffeners and skirt supports for vertical pressure vessels are discussed. The perpendicular projection of rings should be kept to a min because of temp differences, and are preferably located inside the vessel. If fitted externally, insulation is desirable.

The thickness of skirt supports should be kept to a min consistent with structural stability and stress requirements. A. R. H.

110. Oil tanker progress. R. Hammond. *Petroleum*, Nov. 1951, **14** (11), 289–92.—A review of recent developments and trends in oil-tanker construction. It includes brief descriptions of the *British Bulldog* (British Tanker Co. Ltd.), a typical example of a recent oil tanker; the *Jahra*, a typical "super-tanker," and *Butanias* I and II, the first European lighters specially constructed for the exclusive carriage of liquid petroleum gases. R. E. P.

111. An aircraft fueller. Anon. *Engineer*, 1951, **192**, 474.—A new class of airport fueller, the "Dorset," is being introduced on international airfields for refuelling of de Havilland "Comet" jet airliners and other advanced aircraft. It consists of a five-

compartment aluminium-lined steel tank on a Leyland Hippo chassis, and sixty units have been ordered initially. Fuel capacity is 3000 gal, and max loading rate 200 g.p.m., with provision for defuelling, redistribution of wing fuel load, and receiving and discharging fuel simultaneously. The pumping equipment includes a 10-μ filter and a special pressure-control valve which limits shut-off pressure to 8 to 10 p.s.i. above operating pressure (25 to 50 p.s.i.). A. C.

Refinery Operations

Refineries and Auxiliary Refinery Plant

112. California Refining Company triples throughput capacity of Perth Amboy refinery. C. V. Perrine. *World Petrol.*, 1951, **22** (8), 75–8.—Description is given of refinery expansion amounting to nine units, including twin crude units of 27,500 b.d. each, U.O.P. thermal reformer 10,000 b.d., Houdriflow cat cracker 25,000 b.d. and power plant producing 5000 kW and 300,000 lb steam/hr. In addition there are lead sulphide treater, waste-water separators, salt-water pumping plant, and an alkylation unit. Feature of construction was a 200-ton guy derrick with a 200-ft mast mounted on a 65 ft steel tower, allowing of a wide radius of action. E. B.

113. Newest gas processing plant. D. P. Thornton. *Petrol. Process.*, 1951, **6** (7), 736–9.—The Elk City plant of Shell Oil Co. is described for the production of natural gas, LPG, natural gasoline, and crude oil. Present capacity from the gas condenate field is 100,000 M.c.f/day, which is shortly to be increased to 150,000 M.c.f/day. The gathering system and plant are both described. Separators and case storage tanks are not used, but all wells feed to the plant, where initial separation of crude and gas occurs at 950 p.s.i. A schematic flowsheet is given which includes temp and pressures for design conditions with debutanized gasoline operations. E. J. C.

114. Brazil's first complete refinery. Anon. *Petrol. Process.*, 1951, **6** (6), 629–30.—A general description of a 2500-b.d. combination, topping, and thermal cracking plant near Salvador, Brazil. E. J. C.

115. European refining and crudes from the Middle East. G. Egloff. *Refiner*, 1951, **30** (10), 73–8 (*Nat. Petroleum Assoc. Atlantic City, Sept.* 1951).—Figures are given for the proved reserves, production, and refinery capacities of Persia, Iraq, Saudi Arabia, Kuwait, Qatar, and Bahrein, and also of the demands and refining capacities of European countries. A. R. H.

116. The Barton lubricants plant. Anon. *Petroleum*, Dec. 1951, **14** (12), 332.—A brief description of the reconstructed lubricants plant of Shell-Mex and B.P. at Barton, Manchester. The south side is mainly a 21-million-gal tank farm and constructional and maintenance units, whilst the north side is essentially a factory area producing lube oils and greases. A vertical system is employed for blending; the components are fed by gravity from tanks in a structure 80 ft high into the appropriate mixing tank. The factory is capable of producing 15,000 to 20,000 tons/yr of grease. R. E. P.

117. The Fawley refinery. Anon. *Petroleum*, Dec. 1951, **14** (12), 318–26.—A technical description of the Esso Petroleum Co's £27,500,000 refinery at Fawley, near Southampton, which emphasizes the part played by many British manufacturers and contractors. The refinery will be the largest in Europe; the main units are on stream, but the lube-oil and sulphur plants will not be completed until 1953. The equipment includes a 65,000-b.d. single-stage atmospheric crude distillation unit; a 60,000-b.d. combination atmospheric and vacuum unit; a 37,000-b.d. fluid catalyst plant; and a 3000-b.d. Edeleanu sulphur dioxide extraction unit. Crude input will be about 5½ million long tons a year, the main products being motor spirit, solvent naphtha, white spirit, tractor fuel, turbo-jet fuel, kerosine, high-speed diesel oil, light industrial diesel oil, gas-works gas oil, marine diesel oil, lube oils, light fuel blending stock, bunker fuel oil, asphalt, and LPG. R. E. P.

118. Fawley refinery. H. B. Milner. *Nature*, 1951, **168**, 541.—Describes the construction of the new refinery. Among the units are three fractionating towers, a catalytic cracking plant, a thermal cracking plant for production of high-octane spirit, and a polymerization unit. Associated units include a debutanizer tower, Edeleanu plant for refining kerosine, and a hypochlorite plant. The lube-oil plant includes de-asphalting, phenol extraction, propane dewaxing, and clay treatment processes.

Other products include blown bitumen and anti-corrosion additives for lube oils. Of special interest is a plant with output 12,000 tons/yr for the conversion of H_2S into S of 99·5% purity. H. C. E.

119. The Isle of Grain refinery. Anon. *Petroleum*, Dec. 1951, **14** (12), 328–9.—A description of the A.I.O.C. Kent oil refinery expected to be completed in 1953. Crude charging capacity is 80,000 brl/24-hr day. The final output is estimated as 4,000,000 tons p.a., the principal products being: motor spirit (965,000 tons/yr); kerosine (110,000 tons/yr); tractor vaporizing oil (29,000 tons/yr); gas oil (490,000 tons/yr); fuel oil (1,862,000 tons/yr); lubricants (140,000 tons/yr).

The main process units for the crude-oil section consist of one large crude distillation unit, two vacuum units, and one catalytic cracker. R. E. P.

120. Vacuum's new refinery in Essex. Anon. *Petroleum*, Dec. 1951, **14** (12), 327–8.—Describes the new refinery under construction at Coryton, 25 miles from London, which is due to be completed by Jan. 1953. It is designed mainly for the manufacture of lube-oil stocks, although it will also produce high-grade motor spirit, diesel oils, fuel oil, and petroleum waxes. The final output is estimated as 850,000 tons p.a. R. E. P.

121. Chemical engineering materials of construction. Many Authors. *Industr. Engng Chem.*, 1951, **43** (10), 2193–294.—This review of 1950 literature relating to construction materials of interest to the chemical engineer includes fifteen articles and a tabular compilation (pp. 2283–94) of data on mechanical, thermal, chemical resistance, etc., properties for carbon and graphite, cements, stainless steel and other ferrous alloys, tin and tin alloys, and wood. The fifteen articles cover :—

Alluminium alloys. H. W. Fritts and E. D. Verink, Jr., pp. 2197–9, forty-nine references.
Carbon and graphite. W. M. Gayford, pp. 2199–202, fifty-three references.
Cements. C. R. Payne, pp. 2203–7, 105 references.
Ceramics. J. H. Koenig and N. H. Snyder, pp. 2208–17, fifty-nine references.
Wrought copper and copper-base alloys. C. L. Bulow, pp. 2218–26, 162 references.
Elastomers. H. L. Fisher, pp. 2227–35, 161 references.
Fibres. C. S. Grove, Jr., J. L. Vodonik, and R. S. Casey, pp. 2235–8, seventy references.
Iron, mild steels, and low alloy steels. C. P. Larrabee and B. J. Kelly, pp. 2239–41, sixty-nine references.
Nickel and high-nickel alloys. H. O. Teeple, pp. 2242–51, 231 references.
Paint. J. C. Moore, pp. 2251–5, seventy-one references.
Hard rubber. H. Peters, 2256–6, forty-five references.
Stainless steels and other ferrous alloys. W. A. Luce, pp. 2258–71, 328 references.
Tin and its alloys. R. J. Nickervis, pp. 2272–5, 147 references.
Wood. A. J. Stamm, pp. 2276–9, eighty-three references.
Less common metals [titanium, zirconium, tantalum, molybdenum, and noble metals]. D. B. Broughton, pp. 2280–2, fifty-one references. E. J. C.

122. High pressure water + blasting sand = cleaner heat exchangers. W. C. Uhl. *Petrol. Process.*, 1951, **6** (1), 22–5.—A description, with pictorial flow diagram and photographs, is given of the hydro-sand cleaning unit which was put into service in March 1950 at the Esso Standard Oil, Baytown, refinery. It has now largely replaced all other methods for cleaning heat exchangers. High bundle penetration and smoother surface achieved than with dry sand. The sand loss in the closed recycling system is 10% or less. Sand flow used through the jet is 100 lb/min, water flow 30 g.p.m. at 1200 p.s.i. D. W. F.

123. Three ways to clean exchangers. C. J. Wenger. *Petrol. Process.*, 1951, **6** (2), 150–3.—Turbine cleaning of heat-exchanger equipment has been largely replaced by other methods at a Shell oil refinery. Insoluble scales are removed by jetting with high-pressure water or high-pressure water and abrasives. Five different nozzles are used, one of which, a four-nozzle head, is claimed to give a 50% reduction in cleaning time. For hard deposits dry sand blasting is used. Metal loss is negligible with proper handling of the nozzle. Chemical cleaning of equipment is expected to increase with the completion of a portable cleaning unit, consisting of two trailer-mounted 900-gal tanks. Inhibited HCl is used for water scales, chromic acid and detergents for oily deposits. D. W. F.

124. How to get what you need when you order heat exchangers. D. P. Thornton. *Petrol. Process.*, 1951, **6** (10), 1128–32.—Covers performance and construction data required when ordering heat exchangers. The data in each case is split into (*a*) vital information and (*b*) desirable supplementary information. E. J. C.

125. Heat transfer and heat releases in fluidized beds. N. L. Carr and N. R. Amundson. *Industr. Engng Chem.*, 1951, **43** (8), 1856–62.—A detailed mathematical treatment is presented to determine the temperature relationships in a fluidized bed, depending on flow and mixing assumptions. Two cases are studied, first, when conductivity in the solid is a limiting factor, and secondly, where conduction in the solid is neglected. A practical example is given. E. J. C.

126. How to get what you need when you order a valve. D. P. Thornton. *Petrol. Process.*, 1951, **6** (7), 722–4.—A general article dealing with ordering of equipment. Data required when ordering manually operated valves are enumerated under the headings new valves, replacement valves, and spare parts. **6** (8), 844–7.—Data required when ordering valves are listed under the following headings: (*a*) power-operated valves; (*b*) instrument-operated valves; (*c*) pressure and temperature regulators; (*d*) traps and level controllers; (*e*) relief valves; (*f*) check valves. E. J. C.

127. How to get what you need when you order a pump. D. P. Thornton. *Petrol. Process.*, 1951, **6** (9), 974–7.—Covers the information which should be supplied when ordering pumps or spare parts. Factors involved in the choice of the type of pump for a given duty are not included. E. J. C.

128. How to dry process steam. C. D. Crawford. *Petrol. Process.*, 1951, **6** (7), 734–5.—A description is given of the method used to feed steam into crude stills at the Standard Oil Co. (Ohio) No. 1 refinery in Cleveland. Trouble had been experienced with slugs of water entering the stills and constituting a fire hazard. A knock-out drum was replaced with a centrifugal receiver purifier which reduced water content of the steam to 0·5% or less. The device is described, and a schematic diagram is included to show how the purifier is connected up to provide high-pressure steam before this is expanded to 20 p.s.i.g. in turbo-pumps to yield process steam. E. J. C.

129. Centrifugal gas plant compressors. D. T. McDonald. *Petrol. Process.*, 1951, **6** (6), 622–5.—The equipment, installation, and control of a casing-gas compressor station are described. The operating characteristics of the steam-turbine-driven compressors are shown graphically. After two years' operation, actual operating costs are shown to be as economical as was initially anticipated, but it is not yet possible to decide if they are cheaper than reciprocating compressors. E. J. C.

130. Blow-off of flames from short burner ports. C. W. Wilson and N. J. Hawkins. *Industr. Engng Chem.*, 1951, **43** (9), 2129–35.—Authors develop an analytical expression, based on the concept of a "critical boundary velocity gradient" at the limits of stable flame region, which may be used to correlate the occurrence of blow-off on short, cylindrical port burners. The validity of the expression has been confirmed for two fuel gases, for different sizes of burner ports, and for viscous flow. E. J. C.

131. Effect of temperature on carbon steel in refinery vessels being surveyed. V. B. Guthrie. *Petrol. Process.*, 1951, **6** (7), 719–21.—Evidence is reviewed of graphitization of steels used in refinery vessels. It is believed that it is most likely to occur where temps of over 900° F are employed, *e.g.*, in catalytic or thermal cracking units. Graphitization is defined, and examples given of actual specimens which have been found to contain nodular or " bead " type of graphite. Further studies to be carried out are reported. Although little is known about the mechanism of graphitization in detail, it appears that aluminium used to " kill " steel during manufacture may remain dissolved in the steel as metallic aluminium. This is believed to make the steel more susceptible to graphitization. There is no recommendation, however, for choice of steels for high-temp duties. E. J. C.

132. Design of explosion proof motors. J. Z. Linsenmeyer and C. R. Olson. *Oil Gas J.*, 8.11.51, **50** (27), 276.—The classification of hazardous locations according to the National Electrical Code (U.S.A.), and inspection and test of motors submitted to the Underwriter's Laboratories are reviewed. Data available from explosion tests enable reasonable estimates to be made of max pressures anticipated in a motor. Motor casings must be designed to withstand explosive forces.

Consideration is given to metal-to-metal joints, bolted joints, shaft and conductor seals, insulation, and bearings; and maintenance of explosion-proof motors is necessary to keep them safe for many years.

Other types of motors for use with fire-walls are also briefly discussed. G. A. C.

133. High temperature service with austenitic steels. M. E. Holmberg. *Petrol. Process.*, 1951, **6** (3), 256–61. (*Ann. Mtng, ASME, New York City*, Nov. 26–Dec. 1, 1950.)—In the selection of austenitic steels for high-temp duty factors other than tensile, creep, and stress rupture should be considered. In many applications there is no advantage in using stabilized grades, particularly for high-temp service. A spheroidizing treatment is generally effective where stabilized steel is required. Austenitic steels, especially those for corrosive service, should not be subject to stress relieving, as this leads to carbide precipitation, some even with stabilized grades, without any improvement in physical properties. Many failures are attributable to the high coefficient of expansion and low thermal conductivity of austenitic steels, especially where thermal shock and thermal fatigue are involved. Ferritic steels are better used for services involving the latter conditions. Examples are cited with reference to photographs and photo-micrographs of failures. D. W. F.

134. Smokeless flare stacks. W. H. Smolen. *Petrol. Process.*, 1951, **6** (9), 978–82.—Details are given of an experimental flare stack which was used to determine methods of reducing the smoke emitted from flare stacks. Various means were tried, such as water or steam injection, at the top and bottom of the stack. Steam injection at the top of the stack was found to be the most satisfactory way of reducing stack smoke.

The theory of flare operation is covered briefly. E. J. C.

135. Dissolved air flotation separates oil from waste water. N. A. D'Arcy. *Oil Gas J.*, 8.11.51, **50** (27), 319.—Dissolved-air flotation has proved effective in removal of free oil from waste water, but is not so in removal of dissolved organic material, nor emulsified oil. Addition of colloidal floc-forming chemicals to the system effectively removes emulsified and free oil, as well as suspended solids and some dissolved organic elements.

Laboratory investigations must be made before installation of either dissolved-air or colloid-air flotation systems. Initial cost of flotation system is less than that which depends solely on gravimetric separation, and effluent from flotation system is of very high quality.

Size of flotation systems is related to a 0·35 ratio between the cu. ft/min effluent flow and the sq. ft. area of main flotation chamber. A unit treating 37,000 b.d. has a chamber 12 by 31¾ ft. G. A. C.

136. How waste disposal is supervised in a 260,000 b.d. refinery. Anon. *Petrol. Process.*, 1951, **6** (3), 254–5.—The waste-disposal organization in operation at the Baytown, Texas, refinery of the Humble Oil and Refining Co. is reviewed. D. W. F.

137. How self-adjusting skimmers improve operation of waste separators. W. O. Small. *Petrol. Process.*, 1950, **5** (12), 1300–3.—A successful self-adjusting skimmer as installed

in several Esso Standard Oil refineries is described. It consists of a rectangular float with two open compartments on opposite sides connected by channels. The narrower of the two compartments contains the weir, and the oil flowing over this is removed from the second by pump. The whole skimmer is pivoted so that as the rate of removal from the large compartment is varied the weir automatically positions itself to maintain the skimming-pump suction pipe below the oil level. Skimmers of this type have been made in lengths ranging from 5 to 18 ft. Constructional details are given of a 6-ft skimmer. D. W. F.

138. Gold in the plant scrap pile. D. P. Thornton. *Petrol. Process.*, 1951, **6** (3), 251–3.—The procedure adopted by the salvage department of the Texas City refinery of Pan American Refining Corp. for the recovery of discarded material is described. D. W. F.

Distillation

139. Stepwise plate-to-plate computation of batch distillation curves. A. Rose, R. C. Johnson, and T. J. Williams. *Industr. Engng Chem.*, 1951, **43** (11), 2459–64.—Describes the use of punch-card-type calculating machines for the solution of batch-distillation calculations when simplifying assumptions are not applicable. E. J. C.

140. Punched card calculation of six component distillation columns with heat balancing. A. Opler and R. C. Heitz. *Industr. Engng Chem.*, 1951, **43** (11), 2465–71.—Multi-component distillation involves detailed plate-by-plate calculations as the most reliable method for designing continuous distillation columns. A method for performing these calculations with the aid of a punched-card machine is described. It is based on a modified Thiele and Geddes method for preliminary orientation and the Lewis and Matheson procedure for final balance. E. J. C.

141. Effects of pressure drop on distillation plate calculations. A. M. Eshaya. *Industr. Engng Chem.*, 1951, **43** (9), 2153–5.—The effect of pressure drop in distillation columns operating at reduced pressures is discussed. A method is proposed permitting the establishment of an equilibrium curve corrected for variations of pressure throughout the column. This permits more accurate calculation of the theoretical number of plates. The method should be mainly useful to research workers. E. J. C.

142. Graphical treatment of continuous rectification of a partially miscible binary mixture having the azeotropic point in the non-miscible region. F. Neumann. *Erdöl u. Kohle*, 1951, **4**, 561–5.—Continuous rectification of mixtures such as butanol/water and furfural/water can be carried out successfully by a two-column process. The separated components are removed as bottom products, one in each column, and the heterogeneous azeotrope is resolved by separation of combined overhead streams from both columns into two layers in a decanter, which usually requires condensate cooling. Basic method of operation of such a process is described. Max throughput conditions and influence of the decanter on distribution of re-run to the two columns are discussed. Equations are deduced for the operating diagrams and determination of the cut-point; significance of the latter in the calculation of the columns being explained. Load ratio (throughput) is defined as the quotient of the vapour and liquid loads and its relation described to number of plates, specific steam consumption, and to conditions governing the feed, reflux, and distillate. Load ratios of the two columns linked by the decanter are independent of each other. Choice of an economical load ratio and desirability of reflux pre-heating are considered. P. V.

143. An algebraic representation of vapour–liquid equilibria. W. H. Prahl. *Industr. Engng Chem.*, 1951, **43** (8), 1767–70.—An algebraic method is presented for defining the relation between y, the mole fraction in vapour phase, and x, the mole fraction in liquid phase, for binary systems. It uses a general equation,

$$y = \frac{Cx(A - x)}{Cx(A - x) + (1 - x)(B - x)}$$

where A, B, and C are constants derived by a graphical procedure.

It is claimed that the method gives a quick and accurate manner of determining the number of theoretical plates, and can be used for intra- and extra-polation of experimental x–y curves. E. J. C.

144. How to estimate plate efficiency. I. Rigorous approach to the plate efficiency correlation. J. C. Chu. *Petrol. Process.*, 1951, **6** (1), 39–48.—A rigorous method is derived for evaluating local Murphree plate efficiencies in a binary or multicomponent-system fractionating column, based on the method of Geddes (*Trans. Amer. Inst. Chem. Engrs*, 1946, **42**, 79). The overall mass-transfer coeff in Geddes's relation is split up into the gas and liquid film coeffs, these being determined by the relations proposed by the Chilton–Colburn analogy. For vapour diffusivity Hirschfelder's correlation, based on Chapman's general theory, is recommended, together with Wilke's correlation for liquid diffusivities. For determining bubble size, relations are given for streamline and turbulent vapour conditions, thus enabling the velocity of rise of the bubble to be found. Relations and solutions as given by Lewis are used to convert local to point efficiencies for three particular conditions of vapour and liquid flow on the plate. Thirty-four papers referred to. D. W. F.

145. How to estimate plate efficiency. II. Approximate method for estimation of overall plate efficiency. J. C. Chu, J. R. Donovan, B. C. Boswell, and L. C. Fuhrmeister. *Petrol. Process.*, 1951, **6** (2), 154–7.—An empirical equation is presented for the estimation of overall plate efficiencies of commercial bubble-plate fractionating columns. A relation is also given for bubble-plate absorbers. It differs from previous correlations in that the terms slope of the operating line and effective slot submergence are included. The experimental data drawn upon for the evaluation of the empirical constants are fully presented. The applicability of the equation is restricted to the range covered by the data, viz., L/V ratio 0·4 to 8; effective submergence not greater than 1·5 inches. D. W. F.

146. Pressure drop through bubble caps. Pt III. J. L. Huitt and R. L. Huntington. *Refiner*, 1951, **30** (10), 153–4.—(See Abs. Nos. 2117 and 2530, 1951.) The final article in a series dealing with a study of the pressure drop in bubble caps. The application of the equations developed is discussed. It is concluded that there is a minimum length of slot opening below which the vapour will not pass, and this is a function of the slot width and the surface tension. Surface tension does produce a pressure drop. The equations developed can be used for analysing problems of thermal functioning of bubble caps and trays. A. R. H.

147. Making a profit on asphaltic specialities. J. G. Reidel. *Oil Gas J.*, 25.10.51, **50** (25), 94.—The operation expansion from processing 2500 b.d. of high-asphalt crude to 9500 b.d. at the Kerr-McGee Cosco refinery, Okla, is described.

A vacuum column equipped with a bottom-stripping section and operated with intermediate reflux as well as regular top reflux was installed.

Some two dozen asphalt specialities are produced from the flexible operation, including cutbacks, road asphalts, paints, roof coatings, and pipeline wraps. G. A. C.

148. Problem of the losses of liquid fuels. A. Richter. *Nafta*, Oct.–Nov. 1950, **6**, 317–22.—Causes are mainly such as can be dealt with by saturating the atmosphere above crude oil with C_3 to C_5 fractions and circulating it without losses. In transport all cisterns must be air-tight. M. S.

Absorption and Adsorption

149. Packaged gasoline plant processing 5,000,000 cu. ft. of gas daily. T. P. Cook. *Oil Gas J.*, 25.10.51, **50** (25), 96.—The unit in the West Edmond field, Okla, of the Westoc Gasoline Co. is described, and operating conditions given. Control system is automatic; and whole plant could be easily transported to new location with only loss of concrete foundation and cooling-tower pit. G. A. C.

150. Modern trends in gas plants for catalytic cracking units. F. E. Gilmore and R. D. Bauer. *Refiner*, 1951, **30** (9), 196–217. (*Amer. Inst. Chem. Engrs Mtg, Kansas City*, May 15, 1951.)—Results of computations to compare the effects of absorption

temps and pressures, the number of theoretical plates in the absorber, the use of de-ethanizing absorbers, the uses of sponge-oil absorption to prevent losses of lighter absorption oil components to residue gas and the utilization of various refinery streams as absorption oil are presented. (Authors' abstract.)

A flow diagram is presented representing the latest trends in gas plants of this type. It is designed to recover 90% of the total C_3s with a negligible loss of gasoline components to the fuel gas. Features of this design are the use of a de-ethanizer absorber, a low-mol.-wt. primary lean oil, adequate sponge-oil facilities, and the elimination of all recycle-gas streams from the fractionation equipment. A. R. H.

151. Study of absorption equations is key to improving light ends recovery. A. M. L. Kube. *Petrol. Process.*, 1951, **6** (2), 138–43.—Previously presented before Amarillo, Texas Regional Meeting, Nat. Gas. Assoc. of America, Dec. 8, 1950, under title "Observations and problems in recovery and retention of light hydrocarbons." The various phases in the recovery of LPG products are discussed with a view to clarifying various points of operation and design conducive to the more efficient recovery of the light hydrocarbons. The Kremser and Brown absorption factor equations are reviewed and employed to illustrate certain effects. Plate versus concentration diagrams for a typical absorption system are also used to illustrate the effects of imposing different tower conditions. The rich oil distillation and fractionation system and capacity are covered. D. W. F.

152. Selective adsorption computations with digital computers. A. Rose, R. L. Lombardo, and T. J. Williams. *Industr. Engng Chem.*, 1951, **43** (11), 2454–9.—Existing methods for predicting compositions during selective adsorption processes are exceedingly complex for practical multi-component and non-ideal cases. A stepwise, stage-wise method of calculation is suggested for such calculations, and the application of readily available computers is illustrated. (Authors' abstract.) E. J. C.

153. Absorption calculations by punch card calculators. J. W. Donnell and R. W. Draper. *Industr. Engng Chem.*, 1951, **43** (11), 2449–53.—A method is described which permits absorption calculations to be carried out by a calculating machine. E. J. C.

Solvent Extraction and Dewaxing

154. Newest solvent lube plant—a plant description. G. L. Lupfer. *Petrol. Process.*, 1951, **6** (1), 34–8.—A brief description is given of the new lube-oil treating plant which was put on stream during the summer 1950 at the Lima refinery of Standard Oil Co. (Ohio). With a capacity of 1800 b.d. finished products, the plant includes vacuum distillation, propane deasphalting, furfural solvent extraction, M.E.K. dewaxing, and clay contacting units. Several new developments discussed include the replacement of benzole by toluole in the dewaxing unit, a hot-oil-circulating system for supplying the heat requirements of the furfural and dewaxing units, and a pneumatic clay feeder in the contact unit. D. W. F.

155. Extraction in a pilot-unit spray tower. C. J. Geankoplis, P. L. Wells, and E. L. Hawk. *Industr. Engng Chem.*, 1951, **43** (8), 1848–56.—It is pointed out that data available for spray-type extraction towers are generally only applicable to a specific column. Work is reported which was carried out on pilot plant in which the number of jets from the nozzle could be adjusted to give constant bubble size regardless of flow. The system used was toluene–acetic acid–water.

A method of internal sampling was employed to obtain internal mass-transfer coefficients and to locate end effects. It was stated that analysing the internal operation of different towers with different systems should lead to a generalized correlation of spray-tower extraction data. E. J. C.

156. Modified Edeleanu process for recovery of aromatics. Anon. *Refiner*, 1951, **30** (9), 237–8.—A modification of the Edeleanu process, making it possible to exclude paraffinic and naphthenic hydrocarbons from the SO_2 extract, is described. It involves washing the SO_2-extract at 0° to −20° F with a selected kerosine cut for the recovery of benzene from naphtha fractions or a gas-oil cut if it is desired to produce practically pure aromatics from a kerosine fraction. A. R. H.

157. Notes on dewaxing. Pt I. Plant capacities and yields set by operating techniques. P. Zurcher. *Refiner*, 1951, **30** (9), 119–24.—A detailed discussion of the operation of the pressing process. Many puzzling deviations from normal operation may occur in pressing and sweating, the main causes being: (1) changes in crude-oil supply; (2) stratification of crude oil in storage; (3) time elapse between distillation and dewaxing; (4) fluctuations in still operations, affecting distillations range, visc, and resin content of the wax distillate; (5) defective equipment; (6) lack of supervision; (7) atmospheric conditions.

These factors are discussed separately with the presentation of properties and yields. It is shown that the yield of scale wax is increased as the oil content is decreased.

Increased yields of slack wax were obtained by the use of copperized blankets on the presses, 44·5% compared with 20·8% with white blankets. The press cycle is also reduced, as they remain in good condition longer than the white ones, an increase in production of 4·4% was obtained. A. R. H.

Cracking

158. Many new catalytic processes now available. Anon. *Refiner*, 1951, **30** (9), 183–93.—A review of recent developments in catalytic reforming processes including U.O.P. Platforming, Atlantic catalytic reforming, Houdriforming, Thermofor catalytic reforming, and fluid hydroforming. Details of charge and products are given. A. R. H.

159. Catalytic cracking more attractive than ever with new design improvements. Anon. *Refiner*, 1951, **30** (9), 164.—The recent developments in a number of catalytic cracking processes are reviewed. Processes described are: Socony Vacuum Airlift T.C.C. Unit; Hydroflow process; U.O.P. catalytic cracking; Orthoflow fluid catalytic cracking. A. R. H.

160. Factors controlling ageing of cracking catalysts. G. A. Mills and H. A. Schabaker. *Refiner*, 1951, **30** (9), 97–102.—A study of the factors affecting the ageing of clay and synthetic catalysts. The data show that synthetic catalysts give better performance than clay at high temperatures and in the presence of steam. The performance of T.C.C. Filtrol clay was investigated after various pretreatments and in the top and bottom half of the bed. The effects of the presence of hydrogen sulphide, ferric oxide, nickel oxide, vanadium pentoxide, and coppers were studied, and the accumulation of metal on the catalyst correlated against the metal content of the ash in the charge.

A table is presented giving the cause, effect, and prevention of temporary and permanent ageing of cracking catalysts. A. R. H.

161. Catalytic reforming developments. Anon. *World Petrol.*, 1951, **22** (8), 72–4.—Five new cat reforming processes have been developed since war to up-grade naphtha O.N. or produce aromatic and a gas base stock. U.O.P. Platforming process now has sixteen plants operating or planned in the U.S. with total capacity of 60,000 b.d. Other plants include new Houdriforming unit of 10,000 b.d. linked with Sun Arosorb (adsorptive fractionation) plant for production of chemical grades of benzene and toluene + avgas blending stock. A general description is given of the processes, two of which are normally regenerative, two potentially regenerative, and one (U.O.P.) non-regenerative; two employ moving bed and three fixed bed. E. B.

162. First "air-lift" T.C.C. unit. D. P. Thornton. *Petrol. Process.*, 1951, **6** (2), 146–9.—Operating conditions and product yields are given for the first commercial T.C.C. unit, embodying the air-lift modification, to go on stream at the Beaumont, Texas, refinery of Magnolia Petroleum Co. Twin independent units of 15,000 b.d. charge each are installed in a single structure. The major changes from the conventional T.C.C. are the relocation of the reactor above the regenerator and the substitution of an air-lift for the bucket-elevator. The regenerated catalyst is lifted to the separator-surge hopper, where the catalyst is separated from air before passing down into the reactor. From the reactor four pipes take the catalyst to the kiln, which is divided into four segments, each with a set of cooling coils. Catalyst loss due to attrition is about 3 to 4 tons/day. D. W. F.

163. Moving bed catalytic reforming. Anon. *Petrol. Process.*, 1951, **6** (6), 601–2.—Brief reviews are presented of the Thermofor catalytic reforming and fluid hydroforming processes from details contained in papers presented at the Third World Petroleum Congress, The Hague, May 28–June 6, 1950. Some data are given. E. J. C.

164. Pyrolytic and catalytic decomposition of hydrocarbons. V. Haensel and M. J. Sterba. *Industr. Engng Chem.*, 1951, **43** (9), 2016–22.—A review of the literature for year ending May 1951. Eighty-six references. E. J. C.

165. Houdriforming. C. G. Kirkbride. *Petrol. Process.*, 1951, **6** (6), 603–6.—A catalytic reforming process is described briefly with reference to an advanced pilot plant. The process utilizes selective catalysts for: (1) dehydrogenation of C_6 ring naphthenes, (2) the dehydro-isomerization of alkyl *cyclo*pentanes, and (3) the isomerization of *n*- and slightly branched paraffins into highly branched paraffins. Little hydrocracking occurs, and pure H_2 (97% +) is produced. Comparative data given for the Houdriforming of "East Texas" naphtha (180° to 370° F), with three different types of catalyst, show that for a given vol % yield of the charge of (C_5 +) in the range 75 to 90%, a higher O.N. (clear) is obtained using the dual-function catalyst. A brief thermodynamic study of the reactions

C_6H_{12} (*cyclo*pentane) $\rightleftharpoons C_6H_6 + 3H_2$

C_6H_{12} (*cyclo*hexane) $\rightleftharpoons C_6H_{12}$ (methyl *cyclo*pentane) is included. The process is non-regenerative. E. J. C.

166. Aromatics by platforming. V. Haensel and C. V. Berger. *Petrol. Process.*, 1951, **6** (3), 264–7.—Work on the production of aromatic concentrates, using the Platforming process, is reported. For the max yield of aromatics, instead of high O.N. fuel production, modifications in charge stock and operation are required. It is suitable for the aromatization of C_5 and C_6 ring naphthenes. Data are presented on the naphthene contents of C_6 and C_7 fractions of various straight-run gasolines. Experimental results are reported on the processing of a 150° to 190° F fraction of a West coast crude in a lab-scale unit. Potential yield of 48% wt benzene obtained. Data are also presented on toluene production runs on the unit of the Old Dutch Refining Co., and for the production of C_8 aromatics, suitable for aviation rich-mixture blending stock, on a pilot plant. D. W. F.

167. Platforming of pure hydrocarbons. V. Haensel and G. R. Donaldson. *Industr. Engng Chem.*, 1951, **43** (9), 2102–4.—The experimental apparatus and procedure are outlined for a method of converting pure hydrocarbons to other hydrocarbons by passing them over a heated platinum catalyst in the presence of hydrogen and at pressures up to 950 p.s.i.g. Temps used were of the order of 450° C.

Data are given for the processing of *n*-heptane, *n*-pentane, cumene, methyl*cyclo*hexane, and mixtures of the latter plus toluene.

The results showed that dehydrogenation, hydrocracking, isomerization, and hydrogenation may occur. For example, cumene yielded propane, benzene, *cyclo*hexane, methyl*cyclo*pentane, toluene, xylenes, isomeric C_9 aromatics, isomeric C_9 naphthenes, C_1 to C_7 paraffins, and unconverted cumene. E. J. C.

168. Vis-breaking high vacuum residue. J. G. Allen, D. M. Little, and P. M. Wadill. *Petrol. Process.*, 1951, **6** (6), 612–15.—Pilot-plant data are tabulated for vis-breaking of high vacuum (0·1 to 0·8 mm Hg absolute) residues obtained from two paraffinic and one intermediate base stocks, to increase the yield of catalytic cracking feed stock. The yields and product properties of the pilot plant are compared with those obtained from a commercial unit, the latter being described with reference to a simplified diagram. An important feature is a new type of radiant heater described in Abs. No. 169. The economics of vis-breaking are also discussed. E. J. C.

169. New radiant-type heater. Anon. *Petrol. Process.*, 1951, **6** (6), 616–17.—A highly efficient furnace is described which employs a flameless ceramic-cup burner as the radiant-heat source, using fuel gas for operation. Dimensions and tube details, etc., are given for a 30 million B.Th.U/hr heater designed for vis-breaking high vacuum residues (Abs. No. 168). This furnace contains fifty ceramic burners on each of the

two side walls in the radiant-heat section, all burners being independently controlled. Higher heat-transfer rates are claimed than for conventional heaters, which results in saving of initial tube costs, reduction in residence time, higher allowable vapour velocities, and low pressure drop. Data are also presented to show that this type of furnace gives better yields of higher O.N. gasoline by thermal cracking of catalytic cycle oil than those obtained by using a box furnace. E. J. C.

170. Continuous contact coking. Anon. *Refiner*, 1951, **30** (9), 218.—A 100-b.d. unit for the continuous coking of heavy residual oils and tars which has been operating for a year is described. The continuous process produces more gas oil and less coke than do the conventional coking methods operating on the same stock. A. R. H.

171. 39% benzene yields from a C_6 straight-run fraction. Anon. *Petrol. Process.*, 1951, **6** (3), 249–50.—Information is presented on a catalytic reforming process developed by Atlantic Refining Co. A 11,000-b.d. unit is to be built at its Philadelphia refinery. Process is essentially similar to others of this type except that a new platinum catalyst is used. No details of catalyst available. The unit is capable of producing an aromatic concentrate containing 39% (vol on feed) benzene from a C_6 straight-run cut feed. Yield and quality data are given for motor gasoline, benzene, and toluene production. A simplified flow diagram is included. D. W. F.

172. Ethylene manufacture by cracking process. Anon. *Refiner*, 1951, **30** (9), 266.—A process for the thermal cracking of ethane and propane to produce ethylene, operated by the Lummus Company, New York, is described. A. R. H.

173. Fuels manufacture at McMurrey refinery. J. C. Reidel. *Oil Gas J.*, 1.11.51, **50** (26), 68.—The fluid catalytic cracking and revamped existing thermal cracking units at Tyler, Texas, for charging of distillates from vis-breaking of 26·6° API East Texas reduced crude and for feed preparation respectively are described. Four tables summarize the design data for the units concerned, and a flow sheet is given. G. A. C.

174. Tide water makes process improvement at Avon catalytic cracking plant. Anon. *Oil Gas J.*, 1.11.51, **50** (26), 50.—Methods resulting in yield of 12,200 b.d. of gasoline fractions, including butanes, from a total feed to the fluid catalytic unit of about 25,000 b.d. at Avon, California, are described.

Improvements included installation of internal cellular stripper in reactor and increase of coke-burning capacity by raising regenerator pressure. Flow sheets are given. G. A. C.

Hydrogenation

175. Hydrogenation and hydrogenolysis. H. W. Fleming. *Industr. Engng Chem.*, 1951, **43** (9), 1978–86.—A review of 170 references, including hydrogenation of petroleum. E. J. C.

176. Polymerization of gaseous olefins to gasoline hydrocarbons on phosphoric acid catalysts. W. Sapper. *Erdöl u. Kohle*, 1951, **4**, 550–7.—Gaseous olefins and corresponding paraffins are found in cracked gases, in primary and cracked products of the Fischer–Tropsch synthesis, and in primary products of the Hydrocol process, the C_2 to C_4 fraction of which consists almost completely of olefins. Polymerization processes embodying phosphoric and sulphuric acid cat with low temp and pressure are used to convert such olefins to gasoline hydrocarbons. Cat polymerization of olefinic gases combined with procedures such as thermal cracking, Fischer–Tropsch, and the Carburol Kogasin-cracking process, give marked improvement in gasoline yields, since about 80% of charge is convertible to high O.N. product. Cat used are reviewed, phosphoric and sulphuric acid on various carriers (*e.g.*, in thin films on non-porous quartz or glass) being widely used. Mechanisms of reactions, effects of pressure and temp, influence of mol size of feed gases, and operating techniques are discussed. Time of contact and cat load are important considerations. Following average yields were obtained on polymerization plant at 30 to 300 atm with Fischer–Tropsch olefins as feed: 29% polymer gasoline (d_{15} 0·711, E.P. 200–210° C, Motor O.N. 81); 62%

propane–butane; 5% polymers of b.p. > 200° C; 4% gases. Polymer gasoline (with inhibitor) was blended with Fischer–Tropsch primary gasoline; propane–butane fraction was used partly as automotive fuel and partly fractionated, propane being sold for domestic use, while butane was used as running-in fuel for aircraft engines. High-boiling polymers were added to Kogasin diesel fuel, whilst the gases were used for process heating. Review is made of present-day selective- and co-polymerization and alkylation processes. Differences are noted between the polymerization of naturally occurring *iso*-C_4 olefins and that of straight-chain synthetic Fischer–Tropsch olefins.
P. V.

Polymerization

177. Polymerization. C. C. Winding. *Industr. Engng Chem.*, 1951, **43** (9), 1997–2006.—377 references are cited in this review of 1950 developments. E. J. C.

Alkylation

178. Alkylation. R. N. Shreve. *Industr. Engng Chem.*, 1951, **43** (9), 1908–16.—A review covering the different catalytic methods of alkylation. 204 references.
E. J. C.

179. Alkylation commands new interest from refiners. W. L. Bolles. *Refiner*, 1951, **30** (9), 150–61.—A survey of the alkylation processes employed in refining. Deals with sulphuric acid and hydrofluoric acid alkylation. A. R. H.

180. How to increase alkylate yields. D. H. Putney. *Petrol. Process.*, 1951, **6** (7), 744–8.—An improved design in tube bundles has been developed for HF and H_2SO_4 alkylation contactors. A U-tube, prime-surface-type bundle, rather than the finned-tube, lance-type bundle; it will remove up to 2½ times the heat for the same mean temperature difference as the conventional design.

Important economic advantages cited are: in existing HF plants capacity can be increased without raising power requirements. In new HF plants savings can be realized in investment and utilities cost for a given capacity, because contactors can be installed horizontally without steel supporting structures and with simplified agitator drives.

In existing H_2SO_4 plants, capacity is increased with a new bundle plus a refrigerant-circulating pump. In new H_2SO_4 plants similar savings can be made as for new HF plants.

Mechanical details of the new design are given as well as the old type, and important processing points are covered. E. J. C.

181. Sulphuric acid alkyation for motor and aviation gasoline. J. W. Jewell and C. C. King. *Petroleum*, July 1951, **14** (7), 177–81.—(*Presented before W.P.R.A., San Antonio, U.S.A.*)—The author describes the recent progress in sulphuric acid alkylation of olefins with *iso*butane. A description of the Kellogg cascade mixer reactor is included, and is compared with older types of reactor.

Contact between *iso*butane, olefins, and sulphuric acid is divided into multiple contacting zones. All the *iso*butane recycle and all fresh acid is introduced at the first zone, whilst fresh feed is divided equally between all zones. Flow of acid and hydrocarbon is in cascade from one zone to the next, maintaining the *iso*butane-to-olefin ratio at the highest possible level. Internal emulsion circulation is used instead of external circulation. Advantages are:

(1) Because of the higher effective *iso*butane–olefin ratio in the reactor, a lower external ratio may be used, therefore saving plant and utility costs.

(2) A material reduction in power requirement for circulating emulsion (uses less than 25% of power requirement of jet reactors) and also reduced load on the auto-refrigeration system.

(3) Effect of acid degradation is minimized in cascade reactor, only acid in the final contacting zone is at the lowest strength as compared with the jet reactor when the total acid inventory is at spent-acid strength.

New applications of the process and a detailed discussion of the economic aspects of sulphuric acid alkylation are included. R. E. P.

Isomerization

182. Catalytic isomerization of isomeric hexanes. B. L. Evering and R. C. Waugh. *Industr. Engng Chem.*, 1951, **43** (8), 1820–3.—An experimental investigation is described which was undertaken to determine the manner and rate of isomerization of the hexane isomers in order to establish optimum conditions for commercial *neo*hexane units. The results show that the methylpentanes are first formed from *n*-hexane relatively slowly, but that equilibrium between 2-methylpentane and 3-methylpentane is rapidly established. *neo*Hexane (2 : 2-dimethylbutane) is difficult to form, but once formed is very stable under isomerization conditions. E. J. C.

Chemical and Physical Refining

183. Selective H_2S absorption—a review of available processes. A. L. Kohl. *Petrol. Process.*, 1951, **6** (1), 26–31. (*Oklahoma State Mtng, Amer. Inst. Chem. Engrs, Tulsa*, Oct. 7, 1950.)—The common gas-treating systems are reviewed with reference to their use for removing H_2S selectively from gas streams containing appreciable amounts of CO_2. The iron oxide dry box, iron soda, and Thylox processes are used where essentially complete selectivity for H_2S removal is required. The sodium phenolate, tripotassium phosphate, and ethanolamine processes are typical of partially selective systems. (Author's abstract.)

Flow diagram and reactions are given for each process described. Tabular and graphical pilot-plant operating data are presented for the comparison of triethanolamine and methyldiethanolamine. The latter appears to be a good selective absorbent with a high acid gas capacity, high H_2S-removal efficiency, and low steam requirement. Twenty-seven references. D. W. F.

184. Chemistry of inhibitor sweetening. R. H. Rosenwald. *Petrol. Process.*, 1951, **6** (9), 969–73.—After a preliminary discussion on inhibitor sweetening, author describes the apparatus, procedure, and results obtained by sweetening *cyclo*hexene to which various amounts of *n*-, *sec*-, and *tert*-butyl mercaptans had been added. Sweetening involved the use of caustic soda, oxygen, and U.O.P. No. 5 inhibitor.

A mechanism is put forward for the reactions taking place. It appears that tertiary mercaptans are less readily oxidized than secondary mercaptans, and the latter less readily oxidized than primary mercaptans in these sweetening processes. E. J. C.

185. The achievements of the refining industry in U.S.S.R. with regard to reduction of conversion losses. J. Kuropieska and S. Sukranowski. *Nafta*, Oct.–Nov. 1950, **6**, 283–5.—Polish crudes are of similar nature to these found in U.S.S.R. With the establishment of a socialist government, Poland can utilize the achievements of the Soviets. After a short historical review of Russian discoveries and inventions prior to 1920 the author goes in greater detail into the achievements of the scientists of the U.S.S.R. in development of the pipestill, aromatization using $AlCl_3$, cat cracking, cat cyclization, two-stage cracking, solvent extraction, utilization of sulphuric acid sludge, and waste from filtration processes. Improvements in quality of products, introduction of catalytic processes into practice, and increase in production of carbon black were achieved by 1950. M. S.

186. Problem of refining losses. S. Sukranowski. *Nafta*, Oct.–Nov. 1950, **6**, 313–16.—Losses are due to the volatile nature of hydrocarbons as well as to the way they are treated. Most of the avoidable losses occur in transport and handling. Refining losses are tabulated and compared. A few methods are suggested for their elimination. M. S.

187. Causes of refining losses and means of their reduction. W. Setkowicz. *Nafta*, Oct.–Nov. 1950 **6**, 322–8.—Causes of losses which fall into five categories are enumerated. Each kind is discussed, describing its causes and methods of prevention. Suggestions for making use of various waste materials, *e.g.*, acid sludge and activated clays are made. M. S.

188. Industrial petroleum chromatography. 2. H. Weil. *Petroleum*, Aug. 1951, **14** (8), 205–10.—The second part of an article which appeared in *Petroleum*, Jan. 1951 (see Abs. No. 1435, 1951). This part reviews the history and technology of the period 1914–26.

In the first period of petroleum chromatography enrichment of the main classes of hydrocarbons present in crudes, etc., was achieved and the spatial concentration of a range of non-hydrocarbon components obtained. Quantitative resolution of petroleum into typical hydrocarbon groups, however, was not effected. Chromatographic theories and procedures assumed usually to be of more recent origin were elaborated, at least in outline, by prominent petroleum chemists. R. E. P.

189. Liquid thermal diffusion—a continuous separation process. A. L. Jones. *Petrol. Process.*, 1951, **6** (2), 132–5.—Thermal diffusion in the liquid state can be applied to the separation of components of equal mass but of different molecular structure. No theoretical treatment. The apparatus used is essentially the same as used for gaseous thermal diffusion, *i.e.*, hot and cold parallel plates forming the walls of a slit for batch work; concentric hot and cold tubes for continuous operation. Variables, as affecting continuous operation, considered are column height, temp level, and gradient. The separations achieved with kerosine, gas oils, furfural-extracted lube oil, heavy stock, and slack wax are reported. It appears to be particularly useful for producing high V.I. oils by a concentration of the paraffinic components in the overhead stream. Other applications are separation of tall oil into saturated and unsaturated fatty acids and vitamin concentration in fish oils. D. W. F.

190. New caustic regeneration process improves mercaptan sulphur removal. O. J. Zandona and C. W. Rippie. *Petrol. Process.*, 1951, **6** (2), 136–7.—The results are presented of operation of a pilot-plant process for the regeneration of caustic with an electrolytic cell. The caustic from the mercaptan absorber passes to the cell, where the nascent oxygen produced oxidizes the mercaptans to insoluble disulphides. After settling, any remaining disulphides are removed in a naphtha absorber. 52 to 81% conversion of mercaptans during regeneration is claimed. In comparison with steam regeneration, greater removal of mercaptans from both gasoline and caustic are reported. Also lower operating costs. Estimated overall cost 1·3 c/brl gasoline. Stronger caustic solution can be used, around 40%, with less corrosion. D. W. F.

191. How to conserve sulphuric acid. W. F. Bland. *Petrol. Process.*, 1951, **6** (8), 865.—Consumption of sulphuric acid by petroleum refiners increased from 11·8% of total U.S. consumption of the acid in 1948 to nearly 14·2% of the estimated 1951 total consumption. As the supply question is not good, uses are suggested for alkylation spent sulphuric acid in refining, *e.g.*, straight-run and cracked naphthas can be treated with spent acid for gum stabilization and sulphur removal; furnace oils can be treated with this acid; lube oils may be treated with spent acid, although unpleasant odours may result which necessitate further treatment. In all, the report covers information provided by twelve leading American refiners. E. J. C.

Special Processes

192. Aromatics by adsorption. V. B. Guthrie. *Petrol. Process.*, 1951, **6** (8), 833–5.—A new process, "Arosorb," developed by Sun Oil Co. is described for recovery of aromatics from suitable feeds. The process depends upon the selective adsorption of aromatics by silica gel, and their subsequent removal by using suitable hydrocarbon streams of different b.p. range to that of the feed. Separation is then completed by distillation.

A plant is to be built to produce 13,000,000 gal/year of benzene, 30,000,000 gal/year of toluene, and 15,000,000 gal/year of xylenes. E. J. C.

193. Arosorb process for the separation of aromatics. Anon. *Refiner*, 1951, **30** (9), 227–30.—The Arosorb process is a commercial development of the analytical method of silica-gel adsorption by the Sun Oil Company to recover benzene, toluene, and xylenes from a 10,000-b.d. Houdriformer. The feed is passed through a bed of silica gel which adsorbs the aromatics, non-aromatics are displaced by butane, the desorbent

and aromatics are then displaced by mixed xylenes, and the cycle repeated. Six silica-gel cases are to be employed, the cycles being staggered to enable continuous operation of the plant. A catalyst life of a year is anticipated from pilot-plant work. Recoveries from 90 to 98% are obtainable with purities ranging from 98 to 90%.
A. R. H.

194. Urea extractive crystallization of straight-chain hydrocarbons. W. A. Bailey, R. A. Bannerot, L. C. Fetterly, and G. A. Smith. *Industr. Engng Chem.*, 1951, **43** (9), 2125–9.—A new process is described which depends upon the selective reaction of urea with straight-chain hydrocarbons for separating *n*-paraffins or olefins from petroleum fractions up to heavy lube oil.

The design and operation of a 2-brl-capacity pilot plant are described with reference to a flowsheet. In brief, an aqueous solution of urea is contracted with the petroleum fraction + suitable solvent (methyl *iso*butyl ketone), care being taken that the urea solution is saturated at a higher temp than the final reaction temp. Crystals of urea–paraffin complexes are removed by filtration, and subsequently heated with aqueous urea solution to cause dissociation, yielding straight-chain hydrocarbons as an oil phase.

It is claimed that this process will be valuable for production of *n*-paraffins, *n*-olefins, reduction of pour point of oils, and O.N. improvement. Typical properties of feeds and products are tabulated for a naphtha, light-stove-oil, and heavy-stove-oil feed.
E. J. C.

195. Countercurrent solid-gas fractionation process. A. F. Reid. *Industr. Engng Chem.*, 1951, **43** (9), 2151–3.—A description is given of the apparatus and principle involved for the separation of mixtures in the solid phase by taking advantage of differential rates of vaporization, condensation, and diffusion of the components. Results are tabulated for the separation of benzene–*cyclo*hexane mixtures. It is suggested that after further work the process may prove economically practical for certain separations. E. J. C.

196. Shell benzene recovery process. Anon. *Refiner*, 1951, **30** (9), 233–4.—A description of the recovery of benzene by extractive distillation with phenol. A. R. H.

197. Water gas from methane. P. W. Sherwood. *Petrol. Process.*, 1950, **5** (12), 1308–11.—The available processes for the conversion of methane to water gas are outlined. The process in general use in the U.S. is steam reforming of methane in the presence of a nickel-base catalyst. The effect of temp and pressure on the steam–methane reaction is considered. Operating conditions and some details are given of the catalytic-reforming and partial-oxidation processes. Examples of these are the process developed by the Standard Oil Company of New Jersey and the Linde–Karwat converter. In the latter process part of the methane is burnt with oxygen to supply the heat for the endothermic steam–methane reaction. By careful selection of the reactants, the product gas can be made suitable for direct use in the Fischer–Tropsch, methanol, or ammonia synthesis, after the removal of carbon dioxide. D. W. F.

198. Production of ethylene from petroleum sources. Pt 1. P. W. Sherwood. *Refiner*, 1951, **30** (9), 220–5.—The first of a series of three articles. Possible sources of ethylene, the chemistry of ethylene production by pyrolysis of hydrocarbons, and the effect of operating variables are discussed. The commercial production by the partial oxidation of ethane is also discussed.

Twenty references. A. R. H.

199. Hypersorption in modern gas plants. C. Berg. *Refiner*, 1951, **30** (9), 241–6.—Processes for the recovery of ethylene from lean gas streams, the recovery of acetylene from air oxidation of natural gas, and the separation of nitrogen from low B.Th.U. content natural gas are described. A. R. H.

200. Dehydrogenation of methyl *cyclo*pentane over chromia–alumina catalysts. H. Heinemann. *Industr. Engng Chem.*, 1951, **43** (9), 2098–101.—A description is given of the method and equipment used to dehydrogenate methyl *cyclo*pentane obtained from a petroleum fraction, in an effort to produce *cyclo*hexene or methyl *cyclo*pentene. These latter chemicals are referred to as mono-olefin, the yield of which varied at atm

pressure with factors such as space velocity, % of benzene in charge, temp of reaction, etc. Data are presented graphically and in tabular form. E. J. C.

201. Shamrock recovers 30 tons of sulphur daily at McKee plant. D. H. Stormont. *Oil Gas J.*, 1.11.51, **50** (26), 63.—Sulphur-recovery plant at Sunray, Texas, processing about 1900 M.c.f. gas daily is described. Gas is burnt in a specially designed reactor furnace, cooled, and passed to a catalyst converter using natural bauxite catalyst and finally cooled and sulphur condensed by contact with recirculated liquid-sulphur stream. Principal equipment is made from low-carbon steel. A flow sheet is provided. G. A. C.

202. Sulphur recovery in Canada at Shell's Jumping Pound plant. Anon. *Oil Gas J.*, 25.10.51, **50** (25), 102.—This plant recovers 30 tons of sulphur daily from waste acid gases produced in treating 30 million cu. ft/day of natural gas.

The H_2S-bearing gas is fed to a specially designed furnace, where all the hydrocarbons are converted to CO_2 and water, and one-third of the total H_2S to SO_2 and water.

Combustion gases are passed through a waste-heat boiler, generating 7000 lb/hr of steam; and combined gas and by-pass streams are passed into an aluminium oxide catalyst converter. Cooled gases pass into the sulphur wash tower, liquid sulphur collecting at bottom, which is drawn off to storage. G. A. C.

Metering and Control

203. Refinery control problems. E. D. Mattex. *Refiner*, 1951, **30** (10), 132. (*Texas A. & M. Symposium on Instrumentation, Houston*, Sept. 1951.)—Process-control problems are treated with the case-history technique to illustrate methods of solution.

Priming facilities for unloading barges, pH control of crude-unit overhead-water draw-offs, steam–air decoking of furnaces, removable thermo-wells and protective guards, regeneration air measurement, flow control of 50° Baumé caustic, dual-range mercury manometer flow meters, and surging of side-stream level controllers are discussed. A. R. H.

204. Are tomorrow's instruments here today? W. C. Uhl. *Petrol Process.*, 1951, **6** (10), 1123–5.—Author reports on the Sixth National Instrument Conference and Exhibit, September 10–14, in Houston, Texas. Main points of interest for petroleum processing are stated to be: (*a*) tendency to make more use of servo-mechanisms; (*b*) increasing application of electricity and electronics in control equipment; (*c*) development of automatic analysers for continuous recording in the monitoring of petroleum and chemical product quality.

Applications of infra-red analysers are quoted, and a " continuous recording electrometric titrometer " for determining mercaptan content of sweetened gasoline is briefly described. E. J. C.

205. Gadgeteering with process control instruments. Anon. *Petrol. Process.*, 1951, **6** (3), 275–8.—Several examples of the unusual applications of standard or modified control instruments to the solving of process problems are taken from a paper entitled " Gadgeteering," delivered by J. M. Jones at a short course on " Instrumentation for the Process Industries " at Texas A. & M. College. D. W. F.

206. Refinery instruments—what makes them tick? Pt 1. Introduction and pressure measurement. C. E. Shannahan. *Petrol. Process.*, 1951, **6** (7), 730–3.—First of a series of articles dealing with measurement of process variables and methods used in controlling them. Author briefly analyses a typical process, and then describes the main pressure-measuring elements—Bourdon tube, helical spring, spiral element, diaphragm-type element, and double opposed bellows—with the aid of photographs. The ranges of these various pressure-measuring devices are classified broadly as follows: 1 inch water to 15 p.s.i.g.—diaphragm, bronze spiral, and Hydron bellows; 15 to 500 p.s.i.g.—Bourdon tube, spiral, and helical; 500 to 10,000 p.s.i.g.—Bourdon and helical; 10,000 p.s.i.g. and over—Bourdon and heavy-duty stainless-steel helical. E. J. C.

207. Refinery instruments—what makes them tick? Pt 2. Flow measurement. C. E. Shannahan. *Petrol. Process.*, 1951, **6** (8), 548–53.—Flow equations are derived by reference to flow through an orifice. Venturi tubes, nozzles, and pitot tubes are briefly discussed by reference to photographs. A typical overall arrangement of the mercury manometer type of flow meter is included, together with details of the float and segmental lever, and pressure-type bearing method for obtaining pen movement. Rotatmeters are briefly mentioned, along with induction bridge and magnetic transmission to the recording mechanism. E. J. C.

208. Refinery instruments—what makes them tick? Pt 3. Liquid level measurement. C. E. Shannahan. *Petrol. Process.*, 1951, **6** (9), 983–6.—Covers the description and operation of the more important type of liquid-level measuring instruments, viz., torque-tube assembly, ball-float type, external-float type, differential-pressure type, static-head type, and the air-bubbler type. E. J. C.

209. Refinery instruments—what makes them tick? Pt 4. Temperature measurement. C. E. Shannahan. *Petrol. Process.*, 1951, **6** (10), 1118–22.—Construction, advantages, ranges, etc., of gas-filled, liquid-filled, and vapour-pressure bulb type of thermometers, as well as pyrometric types of temperature-measuring instrument are described. Radiation pyrometers are not included. E. J. C.

210. How dual installations checked the accuracy of orifice meters. L. V. Cunningham. *Petrol. Process.*, 1950, **5** (12), 1312–17.—For the purpose of checking the flow rates of four light hydrocarbon streams at Baton Rouge refinery, dual-orifice meters were installed in each line. Each meter installation, which contained features not normally considered necessary, was carefully maintained and the obtainable degree of precision and accuracy determined. A sketch is given of a typical installation. Results obtained during four years of use are summarized and also presented in the form of frequency-distribution curves for percentage agreement ratios of the dual installations. Deviations between the dual meters of 1% or less for 85% of the time, or 3% or less for 99% of the time are reported, together with accuracies of 1% or less for 60% of the time, or 3% or less for 99% of the time. D. W. F.

211. Basic orifice factors for heavy wall pipe. J. B. Arant. *Refiner*, 1951, **30** (10), 139.—Basic orifice factors are presented for flange taps for 14-inch standard wall pipe, 8-inch through 16-inch schedule 40 pipe and 2-inch through 8-inch schedule 80 pipe. Calculation methods from AGA Gas Measurement Committee Report No. 2 and appendix. A. R. H.

212. Easier control by graphic panel. J. Staekner. *Petrol. Process.*, 1951, **6** (6), 626–8.—Describes in fair detail a graphic panel used for the control of a 5000-b.d. Houdriflow unit and its attendant poly, gas recovery, and fractionating units. Four references are appended to previous articles on graphic panels. E. J. C.

213. Locating fluidized solids bed level in a reactor. G. L. Osberg. *Industr. Engng. Chem.*, 1951, **43** (8), 1871–3.—A hot-wire method of determining the level in fluidized beds is described. Constructional and performance details of a laboratory unit are given. It is claimed that the method could be used in automatic bed-level control, and should be particularly useful in high-pressure units. E. J. C.

PRODUCTS

Chemistry and Physics

214. Aromatization of hydrocarbons. V. I. Komarewsky. *Chim. et Ind.*, 1951, **66**, 480–7.—Lecture on aromatization of naphthenes and paraffins, reviewing work carried out in the U.S.S.R. (Zelinskii), the U.K. (Rideal), and the U.S.A. (Komarewsky). Zelinskii discovered that certain metal cat (Pt, Pd, Ni) will only dehydrogenate six-membered rings, five- and seven-membered rings being unaffected. This led to the geometrical theory of heterogeneous catalysis, relating cat with adsorbed material

reacting at its surface. Dehydrogenation of naphthenes by Pt, Pd, Ni is accompanied by flat adsorption at cat surface and no formation of intermediary products. If oxide cat (Cr, V, Mo) are used, adsorption is at two points only, and intermediate products are formed. In dehydrocyclization of paraffins these two classes of cat are again active, metals at 300° C, oxides at 450° to 500° C, in the former case there is likewise no formation of intermediate products. There is thus correlation between action of metals and of oxides as cat in dehydrogenation of *cyclo*paraffins and of paraffins, difference between two types of cat being in both cases due to manner of adsorption of reactant on cat surface. Summary of results on pilot-plant aromatization of 70° to 150° C cut from S.R. Pennsylvania gasoline, using various cat, is given. Twenty-seven references. V. B.

215. Equilibrium relations of two methane–aromatic binary systems at 150° F. M. Elbishlawi and J. R. Spencer. *Industr. Engng Chem.*, 1951, **43** (8), 1811–15.—Vaporization equilibrium constants are reported for the binary systems of methane–benzene and methane–toluene at 150° F over a range of pressures from atmospheric to the critical pressure of each system. It is stated that the results correlate well with previous work carried out on the relationship between equilibrium constants and the U.O.P. characterization factor. E. J. C.

216. Calculation of equilibrium gas compositions. F. J. Martin and M. Yachter. *Industr. Engng Chem.*, 1951, **43** (11), 2446–9.—In evaluating the performance of combustion processes it is usually necessary to calculate equilibrium gas-product compositions. This involves a tedious mathematical solution of a system of simultaneous equations. The authors indicate how this can be overcome by using digital computers. E. J. C.

217. Catalytic conversion of a diene into deeply coloured polyenes. B. Eisler, A. Wasserman, F. D. Farnsworth, D. Kendrick, and R. Schnurmann. *Nature*, 1951, **168**, 459–61.—*cyclo*Pentadiene can be converted catalytically in $> 90\%$ yield into a poly *cyclo*pentadiene–trichloroacetic acid adduct (I) which is deep red in colour. I has the composition $(C_5H_6)_rXY$ where $r_{av} = 18 \pm 2$ and XY = trichloroacetic acid. Evidence of the structural formula of I is deduced from its chemical properties. Absorption bands occur between 2500 and 2800 Å; their absence elsewhere in the spectrum (up to 10,000 Å) is attributed to the fact that I is a mixture of homologues.

From a kinetic standpoint the initial rate of monomer consumption in CCl_4 is prop to the monomer concentration and to the square of the XY concentration; r does not depend on the initial concentration of reactants or on the degree of conversion up to 80%. These facts can be interpreted by a chain-polymerization reaction involving intermediates of short life. If the catalyst concentration is sufficiently low, dimerization occurs as the main reaction.

Experiments prove that the groups X and Y in I are respectively CCl_3COO and H, and not CCl_3COOH and Cl. Mol. wt. determinations show that I is strongly associated in benzene. If the benzene solution is shaken with aqueous solutions of salts, acids, or bases, stable viscous emulsions are formed. H. C. E.

218. Catalytic dehydrochlorination of ethylene dichloride. J. C. Ghosh and S. Rama Das Guha. *Petroleum*, Oct. 1951, **14** (10), 261–4.—The dehydrochlorination of ethylene dichloride to vinyl chloride has been studied using various catalysts and elevated temps. A catalyst prepared from active charcoal containing 10% of (SnO_2.$FeCl_2$) yielded 88·2% of vinyl chloride at 500° C, and was the best yield obtained from the six catalysts used. Data are also presented for the chemical equilibrium $CH_2Cl.CH_2Cl \rightleftharpoons CH_2 = CHCl + HCl$ at pressures of 600 to 700 mm Hg and temps 270° to 360° C in the presence of the catalyst. Heat of reaction = 16,240 cal over the temp range 270° to 360° C.

$\Delta F_T = 16{,}440 - 24{\cdot}2T$, therefore temp of neutral equilibrium ($\Delta F = 0$) = 406° C, showing that vinyl chloride is more stable than ethylene chloride above 406° C.

R. E. P.

219. Observation of cool flames with ethylene. J. R. Abbott and S. A. Miller. *Nature*, 1951, **168**, 474.—Mixtures comprising 15 to 68% C_2H_4, 5 to 6% O_2, remainder N_2 spontaneously ignite at 300° C and pressures up to 41 atm to give two types of flame:

I. At 17 to 41 atm a pale-blue flame moves with relatively small velocity (~ 1 cm/sec) through the mixture, associated with a pressure pulse of ½ to 1 atm. Small amounts of peroxide are formed and 50 to 90% of the O_2 is consumed, 21 to 30% being converted to formaldehyde and acetaldehyde.

II. At 25 to 41 atm normal ignition occurs, and a bright-red flame travelling at > 50 cm/sec is seen. The transient pressure rise is *ca* 30 atm. All the O_2 is used, about 17 to 18% being converted to formaldehyde and acetaldehyde. No peroxide is formed, but much soot is produced.

The pressure at which the flame characteristics change depends on the composition of the gas. In some cases the blue flame is followed by a normal ignition after *ca* 1 sec. The blue flame has the characteristics of a " cool " flame, and its formation is attributed to the diluent effect of the N_2. H. C. E.

220. Mutual solubilities of light hydrocarbon-water systems. W. B. Brooks, G. B. Gibbs, and J. J. McKetta. *Refiner*, 1951, **30** (10), 118–20.—Experimental compositions of the co-existing liquid phases of the *n*-butane–water system are presented at pressures from 100 to 10,000 p.s.i.abs. and at temp for 100° and 200° F.

These data are compared with data previously reported in the literature at the same pressure and temp conditions for methane, ethane, and propane. (Author's abstract.) A. R. H.

221. The reaction of dinitrogen tri- and tetr-oxides with olefins. 2-ethylbut-1-ene and dec-1-ene. C. R. Porter and B. Wood. *J. Inst. Petrol.*, 1951, **37**, 388–95.—Liquid-phase reactions of dinitrogen tri- and tetr-oxides to 2-ethylbut-1-ene and dec-1-ene have been investigated to determine whether these reagents are suitable for the characterization of olefins. Tetroxide addition occurred in only two ways, namely (i) dinitro addition to give 1 : 2-dinitro-paraffins and (ii) nitro-nitrite addition to give, after hydrolysis, 1 : 2-nitro-alcohols; both modes of addition occurring in approx equal extent. In the case of dec-1-ene, however, it was not possible to separate the products completely. A. R. W. B.

222. Substitution products of the isomeric thiophthens. The structure of liquid thiophthen (thiopheno-2′ : 3′ : 2 : 3-thiophen). F. Challenger and R. Emmott. *J. Inst. Petrol.*, 1951, **37**, 396–405.—The structure of liquid thiophthen is shown to follow from two observations: (*a*) the nitration of the methyl ketone and the action of acetyl chloride and aluminium chloride on the nitro-thiophthen yield identical nitro-ketones and thence identical oximes; (*b*) oxidation of the methyl ketone to the carboxylic acid, nitration, and decarboxylation yields the same nitro-compound as is formed by direction nitration of liquid thiophthen. A. R. W. B.

223. Volumetric and phase behaviour in the ethane-propene system. R. A. McKay, H. H. Reamer, B. H. Sage, and W. N. Lacey. *Industr. Engng Chem.*, 1951, **43** (9), 2112–17.—The molal volume of three mixtures of ethane and propene was measured at pressures up to 10,000 p.s.i. over the temp range 10° to 400° F. Plotted data include compressibility factor *vs* pressure at different temps; deviations from ideality *vs* mol fraction of ethane at different pressures; the influence of pressure on the composition of co-existing phases. Bubble-point data and properties of the consisting phases for various pressures are given in tables. E. J. C.

224. Limits of flammability of paraffin hydrocarbons in air. M. G. Zabetakis, G. S. Scott, and G. W. Jones. *Industr. Engng Chem.*, 1951, **43** (9), 2120–4.—An apparatus and procedure are described for determining the lower limits of flammability of paraffin hydrocarbons in air at various temps. The results indicate an average decrease of 8% in the lower limits of flammability of paraffin hydrocarbons for a temp rise of 100° C. Lower limits of flammability of twenty-two paraffin hydrocarbons are given for 26° C. E. J. C.

225. Amination by reduction. J. Werner. *Industr. Engng Chem.*, 1951, **43** (9), 1917–19.—A review of sixty-four references. E. J. C.

226. Esterification. E. E. Reid. *Industr. Engng Chem.*, 1951, **43** (9), 1942–7.—Review covering 189 references. E. J. C.

227. Friedel-Crafts reaction. P. H. Groggins and S. B. Detwiler. *Industr. Engng Chem.*, 1951, **43** (9), 1970–3.—A review of forty-seven references. E. J. C.

228. Halogenation. E. T. McBee and O. R. Pierce. *Industr. Engng Chem.*, 1951, **43** (9), 1974–7.—A review, 104 references. E. J. C.

229. Kinetics and equilibia. R. H. Wilhelm and R. K. Tower. *Industr. Engng Chem.*, 1951, **43** (9), 1898–907.—A selected review of literature published from beginning of 1950 to end of May 1951. 218 references. E. J. C.

230. Nitration. W. de C. Carter. *Industr. Engng Chem.*, 1951, **43** (9), 1987–90.—Nitration of hydrocarbons is included in this review. Forty references are appended. E. J. C.

231. Oxidation. L. F. Marek. *Industr. Engng Chem.*, 1951, **43** (9), 1990–6.—Deals with various aspects of oxidation, including oxidation of hydrocarbons, transformer oils, etc. Ninety-six references. E. J. C.

232. Detergency of carbon-black suspensions. Pt III. In toluene. F. H. Garner and A. R. W. Baddeley. *J. Inst. Petrol.*, 1951, **37**, 426–30.—The rates of settling of carbon-black suspensions have been compared after the addition of stearic, oleic, and linoleic acids, and calcium naphthenate, oleate, and linoleate. The acids have practically no effect on the rate of settling, and are not adsorbed significantly on the carbon. The calcium soaps are adsorbed, and the effect on the rates of settling appears to be dependent on the amount adsorbed; calcium linoleate being the most effective soap. A. R. W. B.

233. Introduction to chemical reaction calculations. Pt 2. (reaction kinetics). A. M. Platt and G. L. Farrer. *Refiner*, 1951, **30** (10), 147–52.—Problems involving reaction rates and residence times are discussed, and worked examples with first- and second-order reactions presented. A. R. H.

234. Evaluation of equation of state constants with digital computers. H. W. Brough, W. G. Schlinger, and B. H. Sage. *Industr. Engng Chem.*, 1951, **43** (11), 2442–6.—The authors describe the use of an electronic type of calculator for evaluating the constants of the Benedict equation of state. Seven constants of this equation were calculated for methane and propane. E. J. C.

235. The separation of sulphur compounds by means of mercuric acetate. S. F. Birch and D. T. McAllan. *J. Inst. Petrol.*, 1951, **37**, 443–56.—Aqueous mercuric acetate enables the separation of certain types of sulphides to be readily effected, and also provides a convenient method for separating various types of sulphur compounds present in the lower-boiling petroleum fractions. A general account is given of preliminary results obtained in using mercuric acetate. A. R. W. B.

236. Sulphonation and sulphation. E. E. Gilbert and E. P. Jones. *Industr. Engng Chem.*, 1951, **43** (9), 2022–52.—Although this review covers much work published in connexion with organic chemistry, sulphonation of petroleum and related substances is also dealt with. 397 references. E. J. C.

237. Laboratory engine load controller simulates actual road conditions. Anon. *Petrol. Process.*, 1951, **6** (1), 52–5.—From a paper by A. R. Isitt, M. R. Wall, and A. G. Cattaneo presented at the Nat Fuels and Lubricants Mtng, Soc. of Auto Engrs, Tulsa, No. 9–10, 1950, under the title "An engine dynamometer control for fuel evaluation by simulated road tests." A brief description is given, with block circuit diagram, of an electronic-type dynamometer control which automatically imposes on a test engine a load substantially identical to that which would be encountered if the engine was installed in a car. Fuel and lube-oil tests can be performed by manual or automatic programme control of the throttle. Satisfactory agreement between road and lab rating is achieved. D. W. F.

238. Grease consistometer is modified to operate at constant shear rates, higher viscosities. Anon. *Petrol. Process.*, 1951, **6** (3), 285.—Modifications made to the McKee worker-consistometer enable it to be operated at constant shear rates and extending the visc range. The modifications chiefly concern the driving mechanism. A cam-operated drive, load up to 200 lb, and a screw-operated drive, load up to 1600 lb, are described. Graphical results of tests are given on a lubricating grease at 100° F and a synthetic rubber at 212° F. D. W. F.

239. The work and programme of Sub-Committe No. 4. Gas, diesel, and fuel oil. H. Gardner. *J. Inst. Petrol.*, 1951, **37**, 352.—This committee is engaged in a search for a suitable pure hydrocarbon to replace IP spirit for asphaltene determinations. Methods for the determination of fuel-oil stability have been reviewed, but no generally acceptable test appears to exist. A fresh approach to the problem of determining the viscosity of fuel oil below 100° F is being made, and it is hoped that a simple test for the pumpability of a fuel oil can be devised. A. R. W. B.

240. The work and programme of Sub-Committee No. 2. Crude oil. H. Jagger. *J. Inst. Petrol.*, 1951, **37**, 349.—The evaluation of crude oil has been dealt with in some detail, and the committee is at present concerned with tests which will indicate to what extent a given crude may be expected to give rise to corrosive troubles.
A. R. W. B.

241. The work and programme of Sub-Committee No. 9. Derived chemicals. P. J. Garner. *J. Inst. Petrol.*, 1951, **37**, 357.—The terms of reference and the achievements of the chemical tests panel and the technological tests panel are reviewed briefly.
A. R. W. B.

242. The work and programme of Sub-Committee No. 12. Greases. E. G. Ellis. *J. Inst. Petrol.*, 1951, **37**, 358.—A brief account is given of the matters which the three panels of this committee have dealt with in the past, and the programmes upon which the panels are at present working are discussed. A. R. W. B.

243. Recent developments in test methods for the knock rating of aviation gasoline. J. Ogle. *J. Inst. Petrol.*, 1951, **37**, 431–42.—At the beginning of the last war it was realized that the Motor and Aviation octane number of a fuel bore little relation to its performance as measured under rich-mixture conditions on full-scale single-cylinder units, and also that good correlation existed between rich-mixture ratings of fuels determined this way on different types of single-cylinder engines. The development of test methods for rich-mixture ratings which was the outcome of this discovery, is described, and the development of weak-mixture-rating test methods is also reviewed.
A. R. W. B.

244. Measurement of water-vapour content of gases dehydrated with triethylene glycol. J. M. Campbell and L. L. Laurence. *Oil Gas J.*, 18.10.51, **50** (24), 93.—Investigations into the use of the Bureau of Mines dew-point tester in plants where diethylene glycol is employed as a desiccant show that the instrument is inaccurate due to glycol content of condensate forming on mirror.

A table shows comparison of dew-point depression measured by the tester with material-balance calculations on the glycol stream. It is considered that prime error with instrument is human one due to too rapid reduction of mirror temp, use of unpolished mirror, presence of excessive amount of hydrocarbons, and inability to distinguish between a water spot and unstable spot caused by hydrocarbons. G. A. C.

Analysis and Testing

245. Improved testing, research methods featured in Pure Oil's new labs. Anon. *Petrol. Process.*, 1951, **6** (3), 268–71.—The recently opened Pure Oil Co's. centre for research and product testing at Crystal Lake, Ill., is described. The centre contains a pilot-plant lab, special photographic department for recording engine test results and an automotive laboratory with mountings to eliminate vibration, besides administration and chemistry buildings. D. W. F.

246. The vapour pressure of gasoline. G. E. Mapstone. *Refiner*, 1951, **30** (10), 157–9.—A nomograph is presented which will determine the pressure correction to be applied to the Reid vapour-pressure test to correct for initial air-chamber temp, bath temp, and barometer pressure. A relationship between the vapour pressure of a variety of gasoline samples as determined by the Reid method and bath temp has also been derived, and is presented in the form of a nomograph. (Author's abstract.)

A. R. H.

247. Determination of lead tetra-ethyl in gasoline and of sulphur and other heavy elements in petroleum products. L. Robert and C. L. Alexanian. *Rev. Inst. franç. Pétrole*, 1951, **6**, 368–74.—Polychromatic X-rays from tube operating at 25 kV with Cu anti-cathode are passed through a 7-cm cell containing test liquid. Transmitted radiation can be detected photographically with subsequent photometry of film, by Geiger counter or by photo-electric cell. Last two procedures require stabler tube operating conditions than first method. Film darkening (1 min exposure) is linear in range of T.E.L. content 0 to 0·1%. Comparative results are given for X-ray (photographic and Geiger) and chemical methods. X-rays can also be used for detection of metal-containing additives and of S. In all cases a blank sample and a sample containing known proportion of sought material are necessary for calibration. V. B.

248. A test for polysulphides in gasoline. G. E. Mapstone. *Refiner*, 1951, **30** (9), 125.—A colour test for the detection of polysulphides in the absence of T.E.L. is described. Sample is treated with alcoholic silver nitrate diluted with water to 85% alcohol. This dilution eliminates interference by reduction of the silver nitrate by tar acid oxidation products, the presence of polysulphides being indicated by a brown or violet coloration.

Interference is caused by mercaptans, coloured gasoline oxidation products, U.O.P. inhibitors Nos. 1, 4, and 5, and Du Pont copper deactivator. Methods for elimination or reduction of interference by these are given. A. R. H.

249. A method for studying the diffusion of waxes through rubber. L. L. Beat. *Brit. J. Appl. Phys.*, 1951, **2** (8), 237.—The constructional details are given of an instrument for studying the diffusion of waxes through rubber. The work described was started with the object of studying the mechanism of, and the laws governing, the diffusion process. A diagram of the apparatus is given, together with a graph of the relation between penetration depth of the crystal boundary and the time of diffusion. A photomicrograph shows wax crystals in rubber after the diffusion process. Experimental details are outlined, and the results give a relation between concentration and penetration distance from which values of the diffusion coeff can be deduced.

C. N. T.

250. Improved methods for the particle size analysis of fluid cracking catalysts. Pt III. F. E. Ivey, Jr. *Refiner*, 1951, **30** (9), 127–32.—(See Abs. No. 2191, 1951.) Screen analyses of powdered catalysts may be predicted from elutriation tests by the equation

$$R = 1{\cdot}854 - 0{\cdot}982\ \rho_E$$

where $\rho_E = \dfrac{K\,100 - A}{100 - B}$

R = density ratio, *i.e.*, the ratio of the particle dia taken from the screen-analysis curve at the weight % level of the elutriation 80-μ fraction, to 80 μ.

ρ_E = elutriation density g/cc.

K = compacted density g/cc.

A = total volatile content of the untested sample in wt %.

B = total volatile content of the 40 to 80 μ fraction in wt %.

A graphical method for the construction of the screen analysis from the elutriation test is described.

Details of modification of the Roller apparatus and the operating procedure used for this study are given. A. R. H.

251. Tests for the consistency of experimental isobaric vapour–liquid equilibrium data. E. F. H. Herington. *J. Inst. Petrol.*, 1951, **37**, 457–70.—The use of isobaric vapour–liquid equilibrium data in the design of precision fractionating equipment is discussed

briefly, and attention is drawn to the lack of a convenient test for consistency of isobaric equilibrium measurements. A test for the consistency of binary data based on the evaluation of the area of the plot of $[\log a_{1\cdot2} - \log(p_1^\circ/p_2^\circ)]$ against x_1 from $x_1 = 0$ to $x_1 = 1$ is presented, where $a_{1\cdot2}$ is the relative volatility ratio of component 1 with respect to component 2 and p_1° and p_2° are the vapour pressures of pure components 1 and 2 at the boiling point of the solution containing a mol fraction, x_1, of component 1. The appropriate extension of the procedure to isobaric ternary systems is indicated.

The method is applied to vapour–liquid equilibrium values for twenty-four systems recorded in the third edition (1950) of the "Chemical Engineers' Handbook," by J. H. Perry, and the data on ten of these systems are shown to be of doubtful consistency.
A. R. W. B.

252. Comment on L. Ubbelohde's method for determination of drop point of greases. R. A. H. Rothe and H. Hamker. *Erdöl u. Kohle*, 1951, **4**, 627–30.—Ubbelohde's method for drop point of grease (DIN 53654) is considered to give insufficient accuracy and reproducibility; modifications are suggested to overcome the following sources of error: (1) subjective errors, such as wrong filling of nipple and poor temp gradient of heating bath; (2) thermometer errors; (3) variations in nipple dimensions; (4) varying materials of construction of nipples; (5) heterogeneity of sample. Experimental results are shown to substantiate the suggestions made. Brass nipples, which give 4° to 5° C lower values of drop point than glass nipples, are considered more suitable. Necessity is stressed for standard pre-treatment of sample; an air-bath is suggested in preference to the present oil-bath. P. V.

253. A vacuum pre-treatment apparatus for filtering, dehydrating and degassing oil. R. G. Martin and C. N. Thompson. *Brit. J. Appl. Phys.*, 1951, **2** (8), 22.—An apparatus for the vacuum pre-treatment of oil has been designed and constructed; the method of using it is described. An assessment has been made of the efficiency of the apparatus which is capable of removing up to 1% added water in one or two treatments, depending on the oil visc, with a consequent improvement in the electrical properties (D.C. resistivity and power factor) of the oils. Loss of volatile oil components has been shown to be very small, and oxidation of the oil during treatment appears to be unlikely. The apparatus has been used to study the rates at which degasified oils of different types re-dissolve air. Under static conditions at 20° C, absorption in the initial stages is very rapid, becoming more gradual later. The rates of re-solution of air between 1 and 6 hr are in the inverse order to the visc of the four oils examined. C. N. T.

254. Engineering fundamentals. Anon. *Oil Gas J.*, 18.10.51, **50** (24), 149.—No. 464 of this series deals with measurement of specific gravity of volatile fluids under high pressure, by means of a 1000 c.c. pyknometer. The specific gravity is obtained, after corrections, from difference in balance weights between instrument filled with liquid at operating conditions and with air or dry gas. G. A. C.

Crude Oil

255. Refining properties of new crudes. Pt 5. Characteristics of crude oils from the "Golden Trend" in Oklahoma. N. A. Smith, H. M. Smith, and C. M. McKinney. *Petrol. Process.*, 1951, **6** (9), 987–90.—Tabulated data are given for thirty-six Oklahoma crudes and compared with analyses of crude oils from older mid-continent fields. A summary is given of the general characteristics of products obtainable from these new crude oils. E. J. C.

Engine Fuels

256. Motor fuel volatility trends. W. M. Holaday and D. P. Heath. *Petrol. Process.*, 1951, **6** (2), 158–60. (*Ann. Mtng, Soc. Auto Engrs, Detroit*, Jan. 8–12.)—The increasing use of cat cracking and cat reforming, which produce gasolines uniform in O.N. throughout their boiling range, will result in motor fuels with higher 90% distillation points. The average 90% point is expected to increase to 350° to 360° F.

With the increasing availability of LPG the initial volatility of motor fuel is likely to rise. These changes in fuel characteristics will require changes in fuel system and engine design over the next ten years, to give engines with high vapour-pressure tolerances and ability to utilize the higher 90% point gasoline. The immediate effect of a military emergency on motor-gasoline volatility, due to aviation-gasoline and jet-fuel requirements, is discussed. D. W. F.

257. Road anti-knock performance of motor gasolines. M. L. Alspaugh and C. A. Hall. *Petrol. Process.*, 1951, **6** (8), 854–60.—The importance of evaluating anti-knock performance of fuels under conditions approximating those experienced on the road is emphasized. Various testing methods are listed, together with factors which influence road rating of fuels. These include make of engine, ignition timing, fuel maldistribution, and variations in temp, humidity, and barometric pressure.

The more recent Borderline Procedure for anti-knock rating (CRC Designation F-8A-943) is described as offering a means for studying relationships between ignition timing, engine speed, and knocking tendency. The relationship of results by this test with those by laboratory anti-knock test methods is discussed. Use of the torque-converter type of automatic transmission may or may not change the maximum anti-knock requirements, depending on the engine. (Authors' abstract.)

Data are presented graphically on the variables cited above. E. J. C.

258. Bonus fuels from selective blending. T. H. Pisk and D. MacDonald. *Petrol. Process.*, 1951, **6** (6), 618–21.—The anti-knock qualities of gasolines are discussed with particular reference to improving T.E.L. susceptibility and improving the "fit" of finished gasolines to contemporary engines. The anti-knock properties of various pure hydrocarbons varies with mixture ratio and engine speeds, and their susceptibility to T.E.L. depends upon their molecular structure.

Octane ratings by research and motor methods are not necessarily the same as the octane rating obtained in an engine on a road test. Some gasolines may give higher road-rating octane numbers than laboratory methods would indicate, a fact illustrated by actual road-test data. The effect of engine speed on leaded and unleaded gasoline, with and without the replacement of the entire C_5 fraction (19%) by *cyclo*pentane, is also illustrated. Similar data are also presented for an insensitive, sensitive, and synthetic gasoline, the last giving very good results. In all cases octane rating is reduced as the engine speed is increased from 1000 to 2800 r.p.m. Test details are not given. E. J. C.

259. Production of high octane gasoline components. Pt II. (Processes). A. A. Draeger, G. T. Gwin, C. J. G. Leeseman, and M. R. Morrow. *Refiner*, 1951, **30** (9), 107–11.—Describes some of the major processes now in use for the production of high-octane gasoline components giving data on yields on O.N. improvements.

Processes dealt with: conversion of gas oils by thermal and catalytic cracking; conversion of residuum by thermal cracking, deasphalting following by thermal cracking and coking; conversion of light ends by polymerization and alkylation; upgrading of naphtha fractions by thermal reforming, thermal recracking, catalytic after-treatment such as *iso*forming, hydroforming, super-fractionation, isomerization, catalytic aromatization, desulphurization, triptane production, and hydrogenation of cracked naphthas. A. R. H.

260. Relation of octane number to coal flame formation. M. R. Barusch and J. R. Payne. *Industr. Engng Chem.*, 1951, **43** (10), 2329–32.—An apparatus and experimental method are described for obtaining a cold stationary flame with a view to investigating the relationship between O.N. and the tendency of fuels to produce cool flames. Various primary reference fuels (O.N. 0 to 80) and various pure hydrocarbons were used in the experiment. The distance of the cool flame front from a sintered plate increased with O.N. Data are tabulated and plotted. E. J. C.

261. Jet fuels. Anon. *World Petrol.*, 1951, **22** (8), 90.—Commercial use of jet fuels is still believed to be ten years in future, until which time military specifications will regulate fuel qualities. E. B.

262. Crankcase explosions. Their causes and practical remedies. Anon. *Petrol. Process.*, 1950, **5** (12), 1304–6. (*Condensed from a paper entitled "Crankcase explosions" presented by F. V. Cook at the Petroleum Mech. Eng. Conference of ASME, New Orleans*, Sept. 24–28.)—From tests on diesel, gas, and dual-fuel engine crankcases it is concluded that explosions result from ignited mixtures of vapour and air which fill the spaces between oil-mist droplets in the crankcase. In certain parts of the crankcase it has been shown that the vapour has the same composition as that being compressed in the cylinder. Methods for preventing explosions are reviewed. A relief valve to be fitted on the crank-case doors is described to minimize the effects of an explosion. The valve lifts at 5 p.s.i., and is fully open at 15 p.s.i., reseating rapidly to prevent inlet of air. Differences in opinion are expressed in the condensed report of the discussion. D. W. F.

263. Characteristics and development of naval fuel oils. M. Blanchier. *Trans. Inst. mar. Engrs*, 1951, **63** (10), 203.—Reviews French naval experiences with fuel oils. Fuels with a sulphur content not exceeding 3·5% could be tolerated, but above this figure excessive corrosion of platework occurred and physiological effects of fumes on operating personnel became pronounced. The presence of sodium in the fuel caused fluxing of refractories and scaling of boiler tubes, but this scaling could be minimized by the installation of water sprays in the tube nest. The main source of sodium in the fuel was due to contamination with seawater, which formed emulsions due to agitation of the ship when under way; the problem of breaking up these emulsions was not completely solved. Pumping difficulties were encountered with fuel oil at low temps, and it was discovered that the congealing point depended on the previous mechanical and thermal history of the fuel. Troubles occurred when using mixed fuels from different sources, and the stability of fuel-oil mixtures was being investigated. The presence of sodium in fuel for "Sural" boilers resulted in water-soluble deposits in the turbines of the turbo-compressors. The use of heavy fuel oil in diesel engines was considered possible. Report of the discussion is included. T. T.

Lubricants

264. A lubricants plant. Anon. *Engineer*, 1951, **192**, 437–9.—A description of the Shell plant recently commissioned at Barton, Manchester, for blending, packaging, and despatch of lube oils, and for grease manufacture. Main storage capacity is 21 million gal, with twenty steam-coiled tanks each of 50 tons capacity at 80-ft level for blending plant feed. Oil is piped from these tanks through distribution boxes to a battery of sixteen 20-ton blending kettles. Barrels of solid fats are melted by infra-red heating muffles before transfer to the distribution boxes for blending. Earth and lime treatment may be carried out in the blending kettles, followed by filter pressing. Electrically operated filling machines handle 150 5-gal drums or 500 1-pint containers/hr. Forty grades of lube oils are despatched. Grease-plant autoclaves are heated by circulating oil at 270° C, and the mixing kettles, into which the contents of the autoclaves discharge, are steam heated or water cooled. Certain greases, *e.g.*, aluminium stearate, required rapid static cooling, and water-cooled rollers are used. A lithium-base grease is also produced by the continuous closed method, involving heating a slurry of lithium stearate/oil in a tubular heater consisting of three concentric tubes, the inner and outer tubes carrying hot oil. The slurry at 220° C is fed on to a water-cooled stainless-steel belt, and is scraped off about ¼ in thick after 30 ft of belt travel. This lithium-base grease has a m.p. of about 190° C, and can function at −55° C. A description is given of the film "Grease," which was shown at the opening ceremony of the plant. A. C.

265. Aircraft lubricating grease needs met with synthetic product. R. R. Matthews. *Petrol. Process.*, 1951, **6** (2), 161.—By reference to five of the papers presented at the 18th Ann. Mtng of the Nat. Lubricating Grease Institute, Chicago, Oct. 30–Nov. 1, 1950, developments in the production and utilization of synthetic lubricating greases are reviewed. D. W. F.

266. Structure of aluminum greases. W. K. Dean. *Institute Spokesman*, Sept. 1951, **15** (6), 12–21.—Previous papers and developments on the theory of grease structure are noted, and material there embodied is utilized in explaining the forma-

tion and properties of aluminum greases. Theories of the structure of aluminum grease are considered in some detail, as is the dispersion of aluminum soaps in oils and the development of the soap structure during cooling. On these bases such properties as clarity, syneresis and bleed, behaviour in working, rebodying and dropping points are explained, and the effect on gel structure is outlined, of other grease components such as mineral acids and alkalis, ammonia and the amines, fatty acids and water, with particular emphasis on the properties of the oils used. J. G. H.

267. Discussion of the fundamental equation of viscosity. M. Roegiers. *Scientific Lubrication*, May 1951, **3** (5), 27–9.—A discussion of the validity of the conventional fundamental equation of liquid visc as compared with that derived by the author by physico-mathematical reasoning from the Boltzmann formula. J. G. H.

268. Viscosity index improvers for lubricating oils. W. S. Port, J. W. O'Brien, J. E. Hansen, and D. Swern. *Industr. Engng Chem.*, 1951, **43** (9), 2105–7.—The theory of visc of high-polymer solutions is discussed and its application to V.I. improvers. Experiments showed that polymers and copolymers of vinyl esters of long-chain fatty acids are effective V.I. improvers for lubricating oils. Thus the addition of 5% polyvinyl palmitate (degree of polymerization 400) to an oil A, gave a V.I. improvement from 90·2 to 123·7, and pour point increased from 25° to 40°. E. J. C.

269. Importance of using low viscosity oils. T. C. Thomsen. *Scientific Lubrication*, Mar. 1951, **3** (3), 16, 33.—The tendency towards the use of lower-viscosity oils with high-speed equipment is remarked, and the relations between fluid and boundary lubrication and between visc and oiliness are discussed with examples drawn from textile practice and from automotive engineering. J. G. H.

270. Compressor lubricants—and how to select the right ones. K. L. Hollister. *Petrol. Process.*, 1951, **6** (3), 272–4. (*Amer. Soc. Lubrication Engrs*, Jan. 25, 1951.)—As the result of a comprehensive study of compressor lubrication made by The Texas Co., data are presented on different oils, which were tested for their characteristics of importance in compressor lubrication. These have been found to form a satisfactory basis for the selection of commercial compressor lubricants. Factors to consider in the choice of lubricants where air, petroleum, and other gases are compressed are discussed briefly, also the lubrication needs of special types of compressors. D. W. F.

271. Utility of greases at low temperatures. Anon. *Scientific Lubrication*, May 1951, **3** (5), 25–6.—Details are presented of investigations carried out recently in Sweden, testing the utility of lubricating greases at low temperatures. This is effected by a series of frictional measurements at different temp in an apparatus comprising three single-row ball bearings on a common horizontal shaft with a cooling bath. The two outer bearings, lubricated with the grease under test, are weighted, while the middle one is blind and is used for measuring the temperature. The method of operation is described in some detail, together with the characteristic charts of friction measurement from which the lower limits of grease utility are derived. J. G. H.

272. Lubrication laboratory of M.E.R.O. F. T. Barwell. *Scientific Lubrication*, Apr. 1951, **3** (4), 10–14, 30.—Details are presented of the new lubrication laboratories of the Mechanical Engineering Research Organisation of D.S.I.R. The individual laboratories and their equipment are briefly described, and research in progress is summarized under the heads of hydrodynamics lubrication, boundary lubrication, mixed lubrication, and wear causes and prevention. J. G. H.

273. Oil film thickness indicators. Anon. *Petrol. Process.*, 1951, **6** (10), 1139–41.—It is reported that a sensitivity of 0·00001-inch can be obtained with mutual-inductance types of measuring element for determining oil-film thickness. Two types of coil (air and iron core) systems are used in conjunction with a cathode-ray tube and other auxiliary equipment. E. J. C.

274. Gear reducer upkeep is cut 85%. C. D. Crawford. *Petrol. Process.*, 1951, **6** (10), 1126–7.—Substantial economies in operating cost are reported for a forced-feed lubrication system installed in cooling-tower gear reducers. Details of the arrangement are described with reference to diagrams. E. J. C.

275. Oil tests reveal maintenance requirements. M. B. Flint. *Scientific Lubrication*, May 1951, **3** (5), 10–13, 31.—The place of lubrication in preventive maintenance is remarked, and the functions of lubricating oil in service are enumerated together with the conditions encountered. While the value of used-oil analysis is conceded, stress is laid on the desirability of site testing of oils in service, and details are presented of an oil-inspection kit and of the tests, precipitation number, water and sediment, neutralization number, and viscosity change necessary to measure the four dangerous classes of contaminants. Conditions leading to the formation of gums and lacquers are indicated, and the value of regular oil tests in revealing these conditions is stressed.
J. G. H.

276. Improved worker—consistometer. Anon. *Scientific Lubrication*, May 1951, **3** (5), 16–17.—Recent improvements are described in the McKee Worker–Consistometer designed to measure the effect of mechanical working on the physical properties of lubricating greases. These comprise cam-operated and screw-operated drives, the former with a load range up to 200 lb, and latter up to 1600 lb. Details are given of a typical test run with the equipment. J. G. H.

277. Flow properties of lubricating greases. N. Marusov. *Institute Spokesman*, Aug. 1951, **15** (5), 8–25.—The necessity for absolute determination of grease-flow properties is emphasized, and the theoretical basis of shear and viscosity relations indicated. A new instrument, the Gulf grease viscometer, is described in detail, together with its scope and method of operation. Advantages accruing from the use of this instrument include those to be derived from continuous operation and a repeatability of $\pm 0{\cdot}5\%$. J. G. H.

278. Hydrodynamic lubrication of rotating disks in pure sliding. A new type of oil film formation. A. Cameron. *J. Inst. Petrol.*, 1951, **37**, 471–86.—Classical lubrication theory shows that the formation of an oil film between disks is dependent on the rolling velocity only. Experiments have shown that it is possible to get an oil film under conditions of pure sliding, an effect which cannot be explained by classical lubrication theory. The explanation is advanced that the variation of visc across the thickness of the film introduces an extra term into the standard equation of film lubrication, and this theory is tested numerically. A. R. W. B.

279. Application of radioactive tracers to the lubrication industry. G. M. Leicester. *Scientific Lubrication*, Mar. 1951, **3** (3), 11–14.—The methods of production and properties of radio-isotopes are summarized, and details presented of essential health precautions and methods of measuring radioactivity. Special applications of tracer technique to the measurement of wear in engines, to the investigation of the effect of additives in operation, and to petroleum production and refining are indicated.
J. G. H.

280. Graphite as a lubricant. E. S. Glauch. *Institute Spokesman*, Oct. 1951, **15** (7), 8–15.—The occurrence, properties, and applications of graphite are briefly touched upon. The effect of the type and quality of graphite used for lubrication is discussed, together with its effect as an additive on other lubricants. The special properties of graphite as a bearing lubricant are outlined, and other applications are indicated.
J. G. H.

Bitumen, Asphalt, and Tar

281. Asphaltic emulsions for road and industrial uses. W. Bierhalter. *Refiner*, 1951, **30** (9), 103–6.—A brief description of the preparation and uses of asphaltic emulsions. They are such that they are stable for a period of about eight weeks, and break due to loss of water by evaporation or absorption by the mineral aggregate to which they are applied. Metallic soaps are used as emulsifying agents. Saponification value of fatty oil 150 to 180. Fatty constituent or fat–acid–oil constituent ranges between 0·5 and 1·0% based on finished emulsion. A. R. H.

282. An improved triaxial compression cell for testing bituminous paving mixtures. C. A. Carpenter, J. F. Goode, and R. A. Peck. *Public Roads*, Aug. 1951, **26** (9), 173.—The triaxial compression test for bituminous paving mixtures at its present state of

development offers a relatively simple means of evaluating cohesion and internal friction in bituminous mixtures. The application of information obtained to the investigation of the stability of existing pavements, and to designing new ones of adequate stability, is somewhat complex. Present-day triaxial cells are subject to two criticisms. The first deals with the "fixed-sleeve" type of cell. Although this arrangement is convenient, an indeterminate stress component is thereby introduced into the test results. The second criticism is one of operational significance, and refers to "free-sleeve" cells. Work carried out by the Public Roads Laboratory to eliminate these criticisms is described with details of operating conditions and test results obtained. The inclusion in the apparatus of a cell-guard to prevent injury to the operator in case of accident is also described. R. H.

Special Hydrocarbon Products

283. Aromatization, cracking of hydrocarbon oils. Polycyclic fractions. C. Weizmann, E. Bergmann, H. S. Boyd-Barrett, H. Steiner, and M. Sulzbacher, with J. R. Holker, E. Mandel, J. Porges, and D. Rowley. *Industr. Engng Chem.*, 1951, **43** (10), 2318–21.—Deals with the nature and identification of constituents of the fraction boiling above 200° C obtained by aromatization of hydrocarbon oils described in the following abstract. The presumable mechanism of their formation is also discussed. E. J. C.

284. Aromatizing cracking of hydrocarbon oils. Process data and mechanism. C. Weizmann, E. Bergmann, W. E. Huggett, H. Steiner, and M. Sulzbacher, with D. Parker, K. O. Michaelis, S. Whincup, and E. Zimkin. *Industr. Engng Chem.*, 1951, **43** (10), 2312–18.—A brief review is presented of the mechanism of aromatization of hydrocarbons by cracking, followed by a description of a pilot plant used to investigate the method as an industrial source of aromatics. Information is tabulated on liquid and gaseous yields by aromatization of a naphthenic naphtha (boiling range 110° to 190° C), together with a considerable breakdown of the liquid and gaseous fractions into individual compounds boiling up to about 180° C. The higher liquid fractions have not yet been completely determined, but many of the higher aromatic hydrocarbons have been isolated.

The influence of factors such as temp, materials of construction of cracking tubes, space velocity and operating pressure, etc., is also discussed, as well as the nature of the feed-stock.

Liquid and gas yields are both approx 50% by wt of charge stock for the naphthenic naphtha mentioned above, but these yields do depend upon the initial paraffinicity or aromaticity.

A description is also given of the analytical procedure and treatment of various product fractions. The experimental evidence suggested that the reaction mechanism follows the line of degradation of large molecules to smaller units (*e.g.*, butadiene), followed by diene synthesis to aromatics. Aromatization may also occur as a result of straightforward dehydrogenation of naphthenes. E. J. C.

285. Aromatization cracking of hydrocarbon oils. Influence of the nature of charging stock. C. Weizmann, E. Bergmann, W. E. Huggett, H. Steiner, and M. Sulzbacher, with K. O. Michaelis, F. Popper, S. Whincup, and E. Zimkin. *Industr. Engng Chem.*, 1951, **43** (10), 2322–5.—The effect of feed composition in aromatization cracking of light and heavy naphthas (boiling range 100° to 200° C) is discussed. Experimental data are given. The process was described in *ibid.*, pp. 2312–18, Abs. No. 284. E. J. C.

286. Aromatization cracking of hydrocarbon oils. Conversion of butylene into aromatics. C. Weizmann, V. Henri, and E. Bergmann. *Industr. Engng Chem.*, 1951, **43** (10), 2325–6.—A report is given of experiments carried out in support of the suggestion made in previous papers (*ibid.*, pp. 2312–25) that butadiene plays an important role in aromatization cracking of hydrocarbon oils. The butylene is converted to a liquid product by passing it through tubes packed with either copper metal or certain oxides (*i.e.*, under dehydrogenating conditions). Temp and space velocity were varied, and in all but one case the liquid product was 100% aromatic. Use of propylene also gave 100% aromatic liquids, but carbonization rapidly occurred. E. J. C.

287. Continuous moulding of paraffin wax. L. H. Jennings, J. H. Beard, and V. A. Kalichevsky. *Petrol. Process.*, 1951, **6** (8), 841–3.—A continuous wax moulding system is described in which molten wax is pumped to " tempering " exchangers, where its temp is adjusted to 140° to 160° F, and then to a hopper. The latter is fitted with six adjustable pistons which control the amount of wax allowed to pass into pans on a moving belt. The wax is solidified by passing the pans through a cooling chamber (fourteen passes in all) through which dry, chilled air is circulated at 20° to 30° F. The pans are automatically emptied on to a belt conveyor. Labour requirements per unit wt of moulded wax are one-fifth those for a conventional batch process. E. J. C.

288. New method of slabbing wax. D. P. Thornton. *Petrol. Process.*, 1951, **6** (9), 963–7.—A description is given of a continuous extrusion process in which wax is extruded as a ribbon and cut to the desired length. The wax crystallizer, or extrusion machine, is described with reference to a machine drawing. Briefly, it consists of a figure-eight-shaped barrel in which two screws are rotated in opposite directions. Molten wax enters the barrel at one end and is forced through a die at the other, its state then being plastic. The plastic wax falls on to a conveyor belt, where it is chilled with water to prevent it sticking to the belt.

Careful control is exercised in applying cooling water to the jacket around the screw barrel, and in applying warm water to the extrusion-die jackets. These jacketing systems are described in detail.

The economics of the process are discussed briefly, the chief points being that lower labour requirements and higher outputs are obtained compared with the conventional plate and frame process. E. J. C.

289. Vapour cooling of transformers. H. W. Perry. *Electr. Times*, 1951, **120**, 931–2.—Weight saving of 25 to 33⅓% as compared with oil-cooled units may be effected by continuous spraying of core and coils with liquid fluorocarbons. Advantages include safety, reduced dimensions, possibility of remote location of coolers. Less fluorocarbon than oil for immersion is required, although present cost of former involves greater expenditure. Following experience with two test transformers full-scale unit is under design for N.Y. City. V. B.

290. Water in transformer oil—a critical résumé of the causes and effects of water in oil-filled transformers. M. R. Dickson. *J. Inst. Petrol.*, 1951, **37**, 373–87.—The formation of water in transformer oils by oxidation of the oil and by absorption or abstraction from the atmosphere is discussed. The effects of this water on the breakdown strength, resistivity, and power factor of the oil are described. Many references are quoted. A. R. W. B.

291. Oils for the textile industry. G. Cibula. *Erdöl u. Kohle*, 1951, **4**, 657–60.—Apart from lubricating oils and greases required for general auxiliary machinery such as electrical, steam, and transport equipment, common to most other industries, the textile industry requires petroleum products for: (1) lubrication of special textile machinery, (2) process treatment of the raw materials used. Various highly specialized types of spinning, weaving, and knitting machinery are described, and the desired properties of lubricating oils and greases for their components are specified. The oils used for processing vary according to nature of product: sulphonated vegetable oils are used for treatment of cottons prior to drying, mineral oil products being unsuitable. Wool and jute must be made pliable before spinning; for this purpose emulsified pet products (removable by washing) are better suited than the vegetable oils formerly used. In the brightening of artificial silk, highly refined thin mineral oils are used in conjunction with surface-active chemicals, influencing sheen, loading, etc. Main properties required by the various oils are briefly listed. P. V.

292. Petroleum in the paint, plastics, and rubber industries. A. J. Goodfellow. *I.P. Review*, 1951, **5**, 337.—Recent advances in petroleum technology have resulted in the production of simple hydrocarbon gases, saturated and unsaturated, which form the basis of many synthetic materials for use in the paint, plastics, and rubber industries. Aromatic extracts are used as extenders for drying oils. Volatile oils like white spirit are used as thinners. At the moment about 30 million gal of white

spirit are used, and only 1½ million gal of turpentine. Considerable amounts of plasticizers and solvents from the petroleum industry are now used in the cellulose paint trade. In the plastics industry, benzene (for phenol-formaldehyde), ethylene (for polyethylene), and acetone (for Perspex) are among the raw materials now available from petroleum. The rubber industry has always looked to the petroleum industry for its solvent, and a number of special-boiling-point spirits are supplied. Large quantities of carbon black and of many plasticizers are also used in the rubber industry. R. H.

Derived Chemical Products

293. Aqueous solutions of sodium benzene sulphonate and monosubstituted derivatives. Some physiochemical properties. P. W. Renich and R. Taft. *Industr. Engng Chem.*, 1951, **43** (10), 2376–84.—Properties of sodium benzene-sulphonate and its Cl-, OH-, NH_2-, and CH_3-substituted products were determined at various concentrations in aqueous solutions at 0°, 25°, 45°, and 60° C. These properties included density, visc, surface tension, refractive index, solubility, and conductance. F.p. depressions were also observed. Concentration range was 0·005 up to 1·0 molal. E. J. C.

294. Chemical products containing fluorine. 1. A. R. Barbour and L. J. Belf. *Chem. Prod.*, Nov. 1951, **14** (11), 385–9.—This, the first part of an article dealing with commercial production and applications of fluorinated compounds, commences by classifying these compounds into two groups as follows: (1) Products valued because of high chemical and thermal stability, *e.g.*, fluorocarbons, Freons, and fluorinated polymers, such as "Teflon," "Kel F." (2) Products used as catalysts, *e.g.*, HF, BF_3.

Consideration is then given to the development of two methods for the production of fluorocarbons (compounds containing fluorine and carbon only). The first of these, the Fowler process, involves the passing of hydrocarbon vapour over cobalt trifluoride; the second, the catalytic process, involves the reacting of the hydrocarbons with fluorine in the presence of a catalyst. Properties of fluorocarbons are described, and mention is made of the electrochemical process as a possible advance in preparative technique. Finally, the development of a range of products termed the Freons (a trade name originated by Kinetic Chemicals Inc. to designate the chlorofluoro carbons and chlorofluoro-hydrocarbons developed by them for use as refrigerants) is discussed, with special reference to Freon 12 (dichlorodifluoromethane), the preparation and properties of which are described. M. C. B.

295. The Catarole process and its products. H. Steiner. *Chem. Prod.*, Nov. 1951, **14** (11), 403–7.—An indication is given of the extent of the petroleum chemical industry in Great Britain. Detailed consideration is then given to the products formed in the Catarole process, the unique feature of this cracking process being that it converts the liquid product into one which is entirely aromatic. A typical analysis is given of the gaseous product, and then the chemicals derived from two of the gases formed, namely ethylene and propylene, are discussed. The composition of the liquid product is described, and the applications of the lower-boiling fraction (boiling below 200° C) and the higher-boiling fractions are considered separately. M. C. B.

296. Petroleum refining waste and by-products as raw materials for other industries. K. Baranski. *Nafta*, Oct.–Nov. 1950, **6**, 328–32.—Included in the Six-year Plans are extraction and utilization of all refinery waste. The general outline is to make use of naphthenic acid salts, sulphonated acids and sulphonic acids, also as salts, generally as surface-active agents. Solvent refining provides plasticizers and drying oils. Other uses are to be developed in conjunction with industries interested in new products. M. S.

Coal, Shale, and Peat

297. Improved process developed for retorting oil shale. Anon. *Petrol. Process.*, 1951, **6** (7), 740–3.—A pilot-plant, vertical retort is described for processing oil shale at the U.S. Bureau of Mines experimental oil-shale refinery. Heat is supplied by gas

combustion in the lower part of the retort. It is claimed that the process requires lower capital costs and operating costs than previous retorting methods. Investment and operating costs are tabulated for mining and crushing, retorting, and refining. Refining sequence is described together with product properties. E. J. C.

298. Pyrolysis of coal and shale. C. H. Prien. *Industr. Engng Chem.*, 1951, **43** (9), 2006–15.—This review of the literature from June 1950 covers 277 references. E. J. C.

299. Hydrogenation of shale-oil coker distillate. E. L. Clark, R. W. Hiteshue, H. G. Kandiner, and B. Morris. *Industr. Engng Chem.*, 1951, **43** (9), 2173.—Details are given of the apparatus and procedure used in high-pressure, liquid-phase, catalytic hydrogenation of a shale-oil distillate obtained from coking crude shale oil. The pilot plant is described with reference to a flowsheet. In two runs, at liquid hour space velocities of 0·9 and 1·1, approx 1500 gal of product were obtained (97% recovery). Catalyst activity was satisfactory as evidenced by the reduction of N_2 and S in the resulting products, as well as by the reduction in b.p. Runs in excess of 300 hr were made without regeneration. The product was separated by batch distillation into a bottoms and overhead fraction. The latter is reported as suitable for a jet fuel, and the former as a diesel fuel. E. J. C.

300. Pressure drop in flow of dense coal–air mixtures. C. W. Albright, J. H. Holden, H. P. Simons, and L. D. Schmidt. *Industr. Engng Chem.*, 1951, **43** (8), 1837–40.—An experimental method is described for investigating the pressure drop in a pneumatic system used to feed pulverized coal to a synthesis gas generator. Experimental and calculated results are tabulated, and empirical equations are derived to express the pressure drop in terms of mass velocity of coal–air mixtures and tube dia for flow in horizontal tubes. E. J. C.

301. Properties of various low-temperature coal-tars. H. Eisenlohr and W. Obenaus. *Erdöl u. Kohle*, 1951, **4**, 557–61.—In Germany low-temp coal carbonization is divided into two main types: (i) indirectly heated furnace processes such as the continuous Koppers and Didier steel-walled furnaces or the batch Krupp–Lurgi furnace with refractory walls, (ii) the "purge-gas," directly heated processes, represented mainly by the Lurgi. Combinations of the two types are also used. Tars obtained from Upper Silesian coal by these methods were analysed and their properties compared. No differences existed between the products of the various type (i) processes; comparison of tars from processes of types (i) and (ii) showed that former yielded less gasoline (E.P. 205° C), similar amounts of middle oil, and less pitch. No differences were established in the nature of the phenolic compounds in the two cases, but neutral oil of coal-tar obtained by the purge-gas procedure had higher H content. P. V.

Miscellaneous Products

302. Detergents for textiles. S. M. Edelstein. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 51.—The article discusses the general properties of detergents—builders, sequestering agents, C.M.C., foaming—and special aspects related to textiles under mill production. Compounded detergents to give both soil removal and oil emulsification are a textile need, and also detergent types with a low exhaustion rate, where continuous processing is used. In evaluating a detergent for the textile mill, its effect on the final properties of strength and handle are most important. Both acetate rayons and wool are sensitive to alkaline solutions, and some detergents are substantive to wool under alkaline conditions. Neutral detergent scouring with synthetics is thus recommended. With cottons and viscose rayon, substantive products are often deliberately used to produce a desired effect on the final handle of the cloth. N. W. G.

303. New and expanded uses of industrial soaps and detergents. J. W. Perry. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 39.—The title of this article is misleading, since the author discusses—after a survey of the theoretical aspects of surface tension, detergency, etc.—two examples where the detergent applications required some special adjustment of conditions to suit the particular industry.

(*a*) Dry-cleaning soaps based on alkyl aryl sulphonates enable water to be dispersed in the bath. The stability of the emulsion was found to be controllable by the addition of inorganic salts.

(*b*) Use of soap as an emulsifier in the polymerization of synthetic rubbers gave trouble, until it was found that diene-unsaturated fatty acid soaps were interrupting the chain-reaction mechanism. The soap specification was then amended accordingly.
N. W. G.

304. Shaving preparations. Pt 1. Lather type products. M. A. Lesser. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 44.—The type of products required for the shaving-preparation market is changing from shaving-soaps to creams, both brushless and lather type. The essential requirements of any preparation are lathering qualities, resistance to drying on the skin, freedom from skin irritation, and softening of the hair by the water in the lather. The soap in the preparation influences the first three requirements only. This part deals mainly with lather-type shaving-creams. In addition to the requirements stated, these products require satisfactory consistency, economic use, good shelf life, and, for commercial reasons, satisfactory packaging. The products are covered by various standard requirements, the American specifications laying down 40% anhydrous soap (as potash soap) min, fatty acid (as stearic acid) 6·5% max, moisture content 50% max. The fatty oil is mainly coco-nut for good lathering with stearic to improve the consistency of the cream, but other oils such as tallow, palm oil, and olive oil are used in blends. Saponification is achieved by mixtures of caustic potash with lesser quantities of caustic soda. Numerous formulæ are quoted and special additives discussed. These include mucilages, sodium alginate, etc., to improve emulsion stability; addition of cetyl alcohol to give smoothness and a pleasant feel to the skin; zinc stearate in small quantities is used for antiseptic purposes; anti-irritants, such as menthol, are added; additives such as alpha-glyceryl stearic acid or synthetic detergents in limited quantities to improve lathering in hard water. A limited discussion of older types of shaving-soaps, sticks, powders, and liquid soaps is included.
N. W. G.

305. Detergents for shampoos. Anon. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 87.—A summary of a paper from Schimmel Briefs No. 197, August 1951. For use as a shampoo, a detergent must have good resistance to hard water, good solubility, pleasant odour, and no effect upon the skin and hair. Foaming is not essential, though favoured commercially. The article recommends detergents with the amide group, three examples being quoted: (*a*) alkylolamides made from coco-nut oil with two equivalents of diethanolamine; (*b*) sulphated alkylolamides; (*c*) derivatives of sulphosuccinamic acid. Representatives of each of these classes are available under various trade names.
N. W. G.

306. Field testing of waxed floors for slip resistance. W. H. Joy, F. J. Pollnow, S. V. James, and C. W. Gurney. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 138.—A symposium discussing the problem of determining whether or not a given floor wax is "safe." A manufacturer, two users, and an insurance company discuss evaluation techniques. All were in agreement that floor waxes can be evaluated only under actual walking conditions, and most measurements involve a technique of "foot evaluation." Attempts have been made to produce some meter to give a quantitative measure. An interesting method is reported, whereby the floor is polished with a two-brush machine with an ammeter in the circuit. The better the slip resistance, the higher the current on the ammeter. The manufacturer used the method of mass observation to obtain information on the properties of newly developed wax formulæ.
N. W. G.

307. Disposable insect cages. P. J. Spear. *Soap Sanitary Chem.*, Oct. 1951, **27** (10), 143.—The development of residual insecticides has created a problem in laboratory technique, in that toxic residues contaminate cages, and decontamination, when this is possible, is expensive. This article gives a description for the manufacture of two disposable cages:—

(*a*) For the exposure of insects to vapours and aerosols, a cage is made with cardboard ends and sides made of tarlatan cloth of uniform mesh. The cardboard ends give shape and rigidity to the cage and supply the support for the door.

(*b*) For the confining of small insects near residual deposits, a cup cage is made from a 5-oz paper drinking-cup with the bottom removed and covered with a layer of cheese-cloth. The cup containing the insects is placed on the surface containing the residues, the cheese-cloth end serving for ventilation and feeding.

N. W. G.

308. Review of pigment progress. W. M. Morgans. *Paint Mnfr*, Nov. 1951, **21** (11), 397.—In the past year there has been an increase in the manufacturing capacity of some of the most important pigments, but pigment supplies have not become more plentiful. Progress in the fields of lead, zinc, cadmium, iron, and other metal pigments is discussed. The decorative and protective characteristics conferred by using metal pigments is particularly noted. The possible utilization of bauxite wastes as a source of pigments has been the subject of several papers, whilst core or coated pigments have also proved of interest. Research on titanium dioxide has continued along the lines of the orthodox aqueous precipitation technique, vapour-phase decomposition, and oxidation of titanium tetrachloride. The after-treatment of pigments has been studied with a view to improving the shelf life of paints containing them, phthalocyanines were particularly considered. Other aspects reviewed include ultramarine colours, anti-fouling pigments, pigment dyes, methods of production, test methods, conditioning of paints, etc. There are ninety-six literature references.

D. K.

309. Role of surface active agents in pigment dispersions. W. Carr. *J. Oil Col. Chem. Assoc.*, Sept. 1951, **34** (375), 400.—A survey is presented of the methods of improving the dispersion of pigments in various types of media and means of controlling the flow properties of the resulting mixtures. The nature and mechanism of some of the forces involved are indicated. Aqueous wetting agents, substances which will reduce the surface tension of water, are discussed. A wide range of compounds is available. The concentration of the wetting agent in water, not the ratio of wetting agent to pigment, is the important factor when determining the quantity of wetting agent to be used. Aqueous dispersing agents function by ionically charging the pigment particles; these agents are limited in number and are all anionic, *e.g.*, sodium salts of condensation products of aryl sulphonic acids with formaldehyde or its polymers. Cationic agents are known, but have little practical importance. The amount of dispersing agent required is based on the weight of pigment to be dispersed.

To obtain a satisfactory pigment dispersion in an emulsion, aqueous pigment dispersion is necessary. Wetting and dispersing agents are needed when starting with the dry pigment powder, but when an aqueous pigment paste is used the need for a wetting agent is removed, though it may be advantageous to use it for other reasons, *i.e.*, to render making of emulsion easier and to prevent transfer of pigment from aqueous to oil phase.

Transfer of pigment from aqueous to non-aqueous system without drying is termed "flushing." An ideal flushing agent should act as a flocculating agent in water, as a wetting and dispersing agent in oil, and have no emulsifying action. One of the most successful flushing agents is based on a triethanolamine ester.

Surface-active agents are thought to improve wetting of pigments by oils by being absorbed on the surface of the pigment particles so that a more easily wetted surface is offered to the oil. The most suitable type of molecular structure is believed to be one that has a polar group which can be absorbed at the particle surface and a non-polar group to offer to the medium.

In non-aqueous media structure formation must occur to prevent settling out of the pigment particles. The author believes that factors affecting structure formation are degree of wetting of particles by media on one side and the force of cohesion between the particles on the other. Surface-active agents are known which act as wetting agents in such media as drying oils, mineral oils, plasticizers, etc. D. K.

310. Dyeing by the hot-oil process. S. H. Williams. *Amer. Dyestuff Rept.*, 1951, **40** (15), 461.—An outline is given of the work leading to the discovery of the hot-oil process, the initial experiments being conducted with medicinal oil. Details of the developed method are quoted, and the types of oil considered satisfactory and unsatisfactory are described. It is concluded that the ideal oil would possess a high flash point, a high b.p., and low visc. It would not dissolve any components of the

dye composition, would have anti-oxidant properties, and would be readily emulsifiable. Oil-consumption figures are stated, and so far oil-recovery systems have not been economically justified. A laboratory procedure for determining the oil-retention of fabrics is outlined. C. N. T.

311. Reinforcing agents and reinforcement. J. R. Scott. *India-Rubber J.*, 1951, **121** (9), 344.—This paper summarizes the views of American rubber scientists and technologists on: (i) current practice in the use of furnace blacks in rubber; (ii) the present position of non-black reinforcing agents; and (iii) the latest ideas and research developments on reinforcement theory. C. N. T.

312. A rapid method for the detection of drier metals in oils and varnishes. R. M. Lavender. *Paint Technol.*, Oct. 1951, **16** (190), 427.—The method outlined consists of precipitating the drier metals with alcoholic oxalic acid and dissolving the oxalates formed in dilute acid. The acid solution is then spot-tested for the individual metals. The method is rapid, and the sample of varnish required is very small. Spot tests recommended by the author for each metal are described, and the smallest amount of metal detectable by the method is stated. Spot tests discussed are borax bead or ammonium thiocyanate in acetone for cobalt; benzidine after lead converted to lead dioxide for lead; catalytic oxidation of manganese salt to permanganate in acid solution or periodate and tetrabase for manganese; ammonium mercurithiocyanate and copper sulphate or potassium ferricyanide and diethylaniline for zinc; ammonium thiocyanate for iron; flame test for calcium; α-benzoin oxime or *o*-tolidine and ammonium thiocyanate for copper; benzidine and alkaline hydroxide or phosphomolybdic acid for cerium; dimethylglyoxime for nickel; alizarin for aluminium; dimethylglyoxime or phosphomolybdic acid or hydrogen peroxide for vanadium. D. K.

313. A carbon black plant. Anon. *Engineer*, 1951, **192**, 537–9.—An illustrated description of the Philblack plant at Avonmouth. Approx 70,000 tons of oil p.a. is to be processed into two grades of furnace black, 30 million lb of " O " grade (particle size 345 Å) and 20 million lb of " A " grade (particle size 510 Å). The oil is a special petroleum fraction, at present imported from the U.S.A., which does not require heating for transport or storage. Oil is preheated and burned under controlled conditions in reactors, and combustion products cooled by water quench and then by atmospheric radiation to about 700° F. Further water quenching to about 500° F is carried out before passing to electrostatic precipitators, cyclones, and bag filters in series. Carbon-black removal in these stages is 30%, 65%, and 5% respectively. The combined blacks with a density of 3 lb/ft^3 are passed to pellet mills to form a dustless final product of density 23 lb/ft^3. A. C.

314. Applications of Epikote resins. E. S. Narracott. *Industr. Chem.*, 1951, **27**, 410–13.—Shell Chemical Corporation manufacture a range of synthetic resins in the U.S.A. from epichlorhydrin and diphenylolpropane, and production of similar materials (Epikote resins) in the U.K. is proposed. Four grades of resins are involved, m.p. ranging from 70° to 150° C and chemical resistance increasing with increasing mol. wt. These resins require curing by cross-linking with other resins such as urea/formaldehyde or phenolic resins to develop their properties, and the final surface-coating films obtained possess high flexibility, adhesion to metals, and chemical resistance. Details of various formulations and conditions for curing are given, a critical stoving temperature of 195° C being necessary. This temperature may be reduced to 150° C by esterification of the resin instead of cross linking, to give very light-coloured esters, but the chemical resistance of such films is inferior to the cured resins. Air-drying may be employed instead of stoving for varnishes or enamels containing these resins, in which case 0·04% of cobalt naphthenate drier is included. A. C.

315. U.S. petroleum industry to become net sulphur producer. Anon. *World Petrol.*, 1951, **22** (8), 65–7.—U.S. sulphur supply and demand position is discussed. Recent price advances to $22/ton will allow S to be produced profitably by oil refineries. Daily capacities of plants producing S from natural gas vary from 30 to 400 tons. Freight charges from producing to consuming areas tend to restrict output to local

requirements. In 1950 six companies recovered 225,000 tons of S from sour oil and gas; 4% of national output and under half of oil industries requirements. A surplus of S should be produced in a few months according to present trends. Girbotol process, now using monoethanolamine, is most popular method of concentrating H_2S, followed by modified Claus process for S production in which part of the acid gas is burned with controlled air to produce SO_2 and by-product steam. Combustion products are mixed with H_2S and passed over a bauxite cat. E. B.

Corrosion

316. Analysis of some corrosion problems in petroleum refineries. J. F. Mason, Jr. *Refiner*, 1951, **30** (10), 124–31; *Oil Gas J.*, 18.10.51, **50** (24), 14. (*Petroleum Mech. Engng Conf., ASME, Tulsa*, Sept. 1951.)—Fifteen case records of actual corrosion problems are presented concerning the initial use of carbon steel for various equipment items and their subsequent failure. Problems include the handling of gas oil, deproparized naphtha, crude oil, spent sulphuric acid alkalate, naphthenic-base crudes, caustic soda and plumbite, furfural, reduced crude vapours, and cracked propane distillate. A. R. H.

317. Corrosion and its control. M. E. Parker. *Oil Gas J.*, 18.10.51, **50** (24), 127; 25.10.51, **50** (25), 137; 1.11.51, **50** (26), 179; 8.11.51, **50** (27), 331.—Pt 32 of this series concerns minimum-cost protection for secondary lines. A magnesium anode is least-expensive type of cathodic protection which can be installed in small, scattered units. A formula gives estimation of probable life of anode; and it is emphasized that complete records should be made.

Pt 33 concerns corrosion records, which should replace the unreliable verbal system. A form which can be mimeographed is illustrated; this deals with pipe inspection, and gives all relevant details, including location, types of joints, soil condition, and nature of corrosive attack.

Pt 34 deals with particular reference to combating corrosion of external surface of well casings where severe corrosive zones lie at great depth.

To deliver a current density sufficient to afford protection to steel pipe lying in very deep and highly corrosive stratum it may be necessary to employ densities up to fifty times amount needed.

In Pt 35 the copper sulphate electrode for measuring potentials referred to a point in or on an electrolyte is described.

This type has advantages over other kinds of electrodes, as the materials for construction and replenishment are readily obtainable, are not expensive, and copper is exceptionally stable in contact with solutions of its own ions. G. A. C.

318. A new inhibitor for sulphide corrosion. J. A. Caldwell. *Petrol. Engr*, March. 1951, **23** (3), B-58.—Desirable properties of an oil well inhibitor are: (1) should be free-flowing liquid or easily dissolved solid; (2) no objectionable odour; (3) non-toxic.

A product termed Corexit, produced by Humble Oil, has shown encouraging results in field tests. Costs of treatment in the field tests varied from \$0·50 to \$1·00/day/well. C. G. W.

319. Corrosion resistance of alloys in amine gas treating systems. F. C. Reisenfeld and C. L. Blohm. *Refiner*, 1951, **30** (10), 107–15. (*Petroleum Mech. Engng Conf., ASME, Tulsa*, Sept. 1951.)—The use is discussed of aluminium and ferrous alloys for the fabrication of amine gas-treating plants in the light of data obtained on such units.

In glycol amine systems heat-exchanger corrosion can be controlled satisfactorily by the use of aluminium 3·5 or 4 to 6 Cr–½ Mo steel for exchanger bundles. Carbon-steel reboiler bundles can be used under favourable conditions, giving a life of 3½ to 4 years. Carbon steel is not entirely satisfactory for stills, and the use of an aluminium or stainless-steel lining in the region of the still defined by the feed tray and the fourth tray below it or such alloys for the entire top section is suggested.

In most aqueous amine installations carbon steel is suitable for heat-exchanger tubes, but where particularly corrosive conditions prevail, carbon steel and 304 stainless steel were not satisfactory. It is suggested that Monel, Carpenter 20, and possibly

316 stainless steels should give satisfactory service in such cases. Reboiler corrosion can be controlled by the use of Monel, Carpeter 20 and 316 stainless steel for reboiler bundles. High metal-skin temps should be avoided. Carbon steel gives reasonable performance in the stills, and 304 stainless steel has given adequate service in a few stills. A. R. H.

320. Vinyl resin coatings. C. C. Munger. *Oil Gas J.*, 18.10.51, **50** (24), 100.—The composition and properties of vinyl resins are given, and their use as coatings for high resistance to humidity, weather, and chemical attack described.

It is essential that a thoroughly dry clean metal surface is prepared before the prime coat, which ties the following coats tightly to the underlying surface, is applied.

Coating thickness is an important point, as is continuity of coating.

Examples of refinery equipment which have been effectively protected by vinyl coatings include interior of caustic-soda tanks, interior of lead-lined gasoline treaters and acid sludge tanks, and interior of pipelines subject to treating liquids. G. A. C.

321. Casing failures traced to bacterial action. R. Sneddon. *Petrol. Engr*, Aug. 1951, **23** (9), B-7.—A series of casing failures has definitely been attributed to the action of sulphate-reducing bacteria. Corrosive fluids, defective steel electric currents, and salt-water migration were definitely ruled out as a cause of corrosion. Recovery of a section of corroded casing showed a corrosion product of composition similar to that reported in cases of corrosion known to be due to sulphate-reducing bacteria.

This view is corroborated by the pH values of mud used. Corrosion only became appreciable when mud was charged from caustic soda–quebracho–sodium silicate to phosphate–quebracho with a reduction in pH from 10 to 8·5. The limits of activity for sulphate-reducing bacteria are 5·5 to 9.

Bacteriological examination of pipe scale, mud cake, and sidewall cores indicated the presence of active bacteria.

In future drilling a high-pH mud will be adopted to prevent bacterial action.
C. G. W.

322. Chromates in cooling water treatment. O. Hahn. *Erdöl u. Kohle*, 1951, **4**, 565–6.—Presence of bacteria was found to be cause of algæ deposits in cooling-tower reservoirs and pump-suction lines. This fouling necessitated quarterly shut-downs for cleaning. Addition of 0·05% K or Na chromate removed practically all the micro-organisms and prevented fouling without causing corrosion. P. V.

Engines and Automotive Equipment

323. Japanese special-purpose engines. L. Keuleyan. *Gas Oil Pwr*, 1951, **46**, 219–21.—The prototype ZB-35 is a two-stroke engine with one cyl of dimensions 150 mm bore and 200 mm stroke, giving a continuous output of 84·5 b.h.p. at 1450 r.p.m. The head carries four exhaust valves, and scavenging air enters through three rows of 85-mm-dia holes in the cyl liner.

Following tests on this engine, a twenty-cyl Vee-type engine was built. Each cyl head contains four exhaust valves and a centrally mounted injection pump and nozzle. The scavenging-air ports in the cyl liner are arranged to eliminate excessive air swirl. The pistons are in two pieces; the Cr-plated steel crown incorporates a combustion chamber, and the body is of cast iron and carries four compression and two scraper rings.

The engine rating is 2000 b.h.p. at 1600 r.p.m. H. C. E.

324. Pressure charging development. Anon. *Gas Oil Pwr*, 1951, **46**, 217.—If the temp of the air in the cyl is decreased at the moment of fuel injection, a greater amount of fuel can be burned, and a higher output attained, without increasing peak temps and pressures. Application of this principle, applied to a series of Nordberg engines of output 425 to 3200 b.h.p., is said to produce a third more power than a conventional pressure-charged engine.

The new system has four advantages: (*a*) air cooling within the cyl is produced by blowing a large volume of air through the cyl; (*b*) the turbo-charger delivers 30% wt

more air by applying a high pressure, thus enabling more fuel to be burned; (c) the intake air is cooled before it enters the cyl; (*d*) by closing the intake valve before the end of the intake stroke the pressure is reduced from 15 to 6 p.s.i. and the air is thereby cooled 50°. The turbo-charger delivers air at 15 p.s.i. and 160° F; this is cooled to 100° F before entering the cyl, and further cooled to 50° F by expansion to 6 p.s.i.

Application of this principle to a Nordberg four-cycle unit of cyl dimensions 13-inch bore and 16½-inch stroke, operating at 450 r.p.m., increased the output from 150 b.h.p/cyl on conventional supercharging to 200 b.h.p/cyl using the new system. H. C. E.

325. New range of Blackstone oil engines. Anon. *Gas Oil Pwr*, 1951, **46**, 213–15.—The engines of this EV series have cyl of bore and stroke 8⅞ and 11½ inches respectively, and at 600 r.p.m. develop 45 b.h.p/cyl when naturally aspirated and 60 b.h.p. when turbo-charged. A power range from 90 to 480 b.h.p. is provided by units of two to six and eight cylinders. The naturally aspirated engine has a b.m.e.p. of 86·1 p.s.i., and fuel consumption is 0·372 lb/b.h.p.hr. When pressure charged the corresponding figures are 115 p.s.i. and 0·365 lb. The specific weight is 85 lb/b.h.p.

The engine accessories, contained in a housing at the end of the engine, comprise lub-oil and water pumps, oil filters, control gear, and instruments.

Two diagrams and four illustrations. H. C. E.

326. New mixing and governing valve. Anon. *Gas Oil Pwr*, 1951, **46**, 228.—This valve is designed to permit adjustments to the gas/air ratio—as for instance when changing from one gas to another of different calorific value—without stopping the engine. It is of the venturi-choke type, and is operated by turning a control hand-wheel. This operates a worm and toothed wheel which turns the threaded adjustable half of the venturi choke and thus alters the annular width available to the gas supply. For large engines each cyl has its separate valve, all, however, connected to a common hand-wheel; in small multi-cylinder engines one valve is provided for all cylinders. H. C. E.

327. Australia's first main-line diesel-electric locomotive. Anon. *Gas Oil Pwr*, 1951, **46**, 244–7.—In this locomotive the power unit is an English Electric SVT type four-stroke pressure-charged Vee engine with sixteen cyl of bore and stroke 10 inches and 12 inches respectively. It develops 1760 b.h.p. at 750 r.p.m. Each of the four turbo-chargers serves four cyl and has an automatic lubricating system independent of the main engine. Accessories include two water-circulating pumps, two lube-oil pumps, and two fuel-transfer pumps. Forced lubrication is employed throughout.

This engine is coupled directly to a traction generator which feeds the four traction motors. Output is varied by variation of engine speed and of excitation of the generator. An auxiliary generator is provided for excitation of the main generator and for supplying current to auxiliary devices.

The method of driving the locomotive is described in detail. Its 1-hr tractive effort is 37,000 lb at 13·5 m.p.h., and the continuous tractive effort is 34,000 lb at 15 m.p.h. The max service speed is 70 m.p.h. H. C. E.

328. Diesel electric shunters for Ceylon. Anon. *Gas Oil Pwr*, 1951, **46**, 239.—The power unit is a Paxman 12RPHX Vee-type engine with twelve cyl each of dimensions 7 inches bore and 7¼ inches stroke. It develops 625 b.h.p. at 1250 r.p.m. with a b.m.e.p. of 110 p.s.i. It is fitted with a Ricardo Comet Mark III combustion system and twin exhaust-gas-pressure chargers. The fuel-pump control is operated by lube-oil pressure, with a device to provide excess fuel on starting. H. C. E.

329. Wallasey's diesel electric ferry. Anon. *Gas Oil Pwr*, 1951, **46**, 236–8.—This ship is powered by Ruston and Hornsby 6VEBXZ diesel engines directly coupled to 300-kW propulsion generators and 50-kW auxiliary generators.

Each of the four four-stroke engines has six cyl, and develops 540 b.h.p. at 500 r.p.m. on the 12-hr rating, and is supercharged. Dry-sump lubrication is employed. The engines are provided with heat exchangers, oil coolers, and thermostatically controlled cooling system, and each unit is almost self-contained. An alarm system operates if the oil, salt-water, or fresh-water pressures fall, or if the temps become excessive.

Each engine is connected in series with a main and an auxiliary generator. There are also two independent auxiliary 50-kW generators, each driven by a Ruston five-cyl 5VPHZ unit developing 80 b.p.h. at 800 r.p.m. H. C. E.

330. A new Burmeister and Wain diesel engine. Anon. *Motor Ship*, Nov. 1951, **32** (380), 312.—Test-bed trials have recently been run in Copenhagen of a large exhaust-gas turbo-charged marine diesel engine. It was originally a normal B. & W. marine-type, uniflow-scavenged, two-stroke, single-acting, exhaust poppet-valve engine, which was taken from the production line and modified for turbo-charging. The six cyl have a dia of 740 mm and the stroke is 1600. An output of 8000 b.h.p. at 108 p.s.i. b.m.e.p. has been developed. By the use of pressure turbo-charging a material saving in weight and space is obtained, as a six-cyl, moderately turbo-charged unit will develop the same output as an eight-cyl engine of the normal non-turbo-charged type. Development work on the new design was undertaken with a large two-stroke, single-cyl unit, built for general investigation, and a smaller engine with four cyl provided with exhaust-gas turbo-chargers.

Experimental work and modifications carried out are described. Curves are given to show fuel consumption, cyl-cover inner surface temp, and cyl-wall temp, for both normal type and turbo-charged engines. U. M.

331. The Standard-Sunbury engine indicator "Mark 6." E. S. L. Beale and R. Stansfield. *Engineer*, 1951, **192**, 215–17; 246–8.—A detailed description of the latest model of an instrument, first developed in 1935, for examining cyclic changes of pressure and recurrent mechanical movements in engines, compressors, etc. Improvements have been effected in design of pick-up units; the pressure pick-up unit of greatest fidelity is an electro-magnetic type using a permanent bi-pole magnet, though this is less sensitive than previous types, and requires higher amplification. In the case of a diesel-engine cyl pressure, at pressures of about half the max, the mean error on the compression and expansion strokes can be represented by a time lag of about 3½° of crank angle for the earlier type of pick-up, whereas the error on the improved bi-pole pick-up is too small to be measured. Amplifier, time base, and cathode-ray tube (single-beam) circuits have been completely redesigned. A simplified circuit diagram of the three-stage signal amplifier is shown, the overall gain of the three circuits being about 200,000. A "divide by two" feature has been added to the time-base circuit, allowing either of the two half-cycles of a four-stroke engine to be chosen at will. Time base is calibrated in degrees, and time markers (1 milli-sec and 10 milli-sec) have been added. Tracings may be made direct off the 6-inch cathode-ray-tube face, or photographic records may be taken. Photographs of typical diagrams are shown. Instrument operates from a 12-V battery, measures 12½ by 17 by 18 inches, and weighs 70 lb. A. C.

Safety Precautions

332. Minimizing pump-station fire hazards. P. D. Phillips and F. O. Stivers. *Oil Gas J.*, 8.11.51, **50** (27), 291.—Causes of fire such as spontaneous ignition, friction, spark ignition, heating equipment, and static electricity are discussed; and a summary of twenty points regarding minimizing pump-station fire hazards is given. G. A. C.

333. Five fire protection lessons proved by tank farm blaze. *Petrol. Process.*, 1951, **6** (10), 1112–14.—In a recent investigation of cause and damage at a tank-farm fire in California, the following conclusions were drawn: (1) use a closed circulating system for blending butane and gasoline; (2) use all-steel construction for new or replacement pipe manifolds in tank blocks; (3) back-weld all threaded steel connexions to prevent their pulling free when exposed to severe thermal expansion; (4) calculate the risks in using jack-knife-type swing joints for earthquake protection—the packing may burn out; (5) maintain API tanks, so that when overpressured they fail at their roof-shell seams without failure of shell or bottom.

The method of fighting the blaze is also recorded. E. J. C.

334. Dangers in handling sulphur dioxide. R. Freitag. *Erdöl u. Kohle*, 1951, **4**, 569, 579.—Dangers inherent in the use of SO_2 in many industries are reviewed. Figures are given for varying effects of different SO_2 concn; as much as 0·12 mg SO_2/litre air can be temporarily tolerated by acclimatized personnel, but individual cases of hypersensitivity can occur. Symptoms of SO_2 poisoning, its possible lethal effects, prevention, and cure of accidental poisoning are discussed. P. V.

MISCELLANEOUS

335. Refinery expansion in Europe. Anon. *Petroleum*, Oct. 1951, **14** (10), 266–70.—Important sections are given of the Second Report on the co-ordination of oil-refinery expansion in Europe released by the Organization for European Economic Co-operation in Paris. The present report describes the main features of the changing economic background since the Oil Committee made its first study of the national refinery programmes. R. E. P.

336. Refining industry's expansion plans. Anon. *Petrol. Process.*, 1950, **6** (6), 607–10.—Details are presented of a plant-construction programme recommended by the U.S. Petroleum Administration for Defence to increase basic refining facilities by more than: (*a*) 500,000 b.d. of crude charging capacity, (*b*) 465,000 b.d. cracking capacity, (*c*) 90,000 b.d. catalytic reforming capacity. A complete list of the projected plants is given, and the estimated cost is $550,000,000. E. J. C.

337. Refining expansion programme growing. Anon. *Petrol. Process.*, 1951, **6** (8), 836.—The U.S. refining expansion is briefly reviewed and projects additional to those listed in *Petrol. Process.*, 1951, **6** (6), 608 (Abs. No. 336) are tabulated. E. J. C.

338. British refinery expansion. Anon. *Petroleum*, Dec. 1951, **14** (12), 316.—A summary of part of the Second Report on the co-ordination of oil-refining expansion in Europe, which has recently been released by the O.E.E.C. in Paris, dealing with the developments now in progress in the oil-refining industry in this country (See also Abs. No. 335.) Internal consumption will increase from 14 million tons in 1949–50 to 17·8 million tons in 1952–53. Exports in 1949 expected to remain steady at 460,000 tons are now envisaged at a level of 2·5 million tons in 1952–53 (including 1·8 million tons of fuel oil).

A map is included showing in schematic form the major projects which are being constructed, as well as the principal refineries already in operation in England and Scotland. Details of crude charging capacity, cracking capacity, major processes, location, date of completion, and final output is given for each refinery. R. E. P.

339. What's ahead for petroleum in the energy markets? C. F. Smith. *Petrol. Process.*' 1950, **5** (12), 1297–9.—(*From "Opportunities for petroleum in the energy markets of the future" the Ann. Mtng API*, Nov. 13–16.)—Total energy requirements in the U.S., expected to increase by as much as a third by 1960, are considered in relation to the part petroleum will have to play. The energy market is classified into two main sections: (*a*) speciality uses, ones in which oil enjoys unique advantages and transportation uses; (*b*) stationary uses such as power generation and heating for which alternative fuels are available. Future requirements of both categories are estimated, assuming prices to remain fairly constant. The relative demand of the former may decline from one-third in 1937 to one-quarter of the total market by 1960 owing to more efficient utilization, such as almost complete replacement of steam locomotives by diesels and more efficient I.C. engines. Stationary uses are expected to increase even faster than in the past, possibly requiring an increase in middle distillate yield to 30%, as against the present 23%. With no change in gasoline yield, this will mean a lower residual yield. Anticipated oil demands will require crude runs of 6½ million b.d., as compared with the present 5·4 million b.d. D. W. F.

340. Ethylene petrochemicals—today and tomorrow: optimism and caution. W. E. Kuhn and J. W. Hutcheson. *Petrol. Process.*, 1951, **6** (10), 1101–11.—The petrochemical industry in America is stated to have grown from an output of 4100 million lb in 1940 to 16,000 million lb to-day. It is predicted that this rate of growth will continue for the next decade. As far as ethylene is concerned, chemicals which can be derived from this source are listed in a table (two pages).

The authors outline briefly the uses of the more important chemicals from ethylene, giving, where possible, annual production figures for the U.S. Thus ethylene oxide, produced from ethylene by direct oxidation or *via* the chlorohydrin, is used alone as a fumigant and as a sterilizer for starch products. It is, however, in the production of ethylene glycol that ethylene oxide finds its greatest use. Main demands for ethylene

glycol are as anti-freeze, but fair quantities are used in manufacture of synthetic fibres, cellophane, paper, leather, explosives, other organic chemicals, etc. Projected demand for ethylene glycol is shown as 797,000 million lb in 1962.

Acrylonitrile, acrylic synthetic fibres, and ethanolamines are mentioned as other main outlets for ethylene oxide. The demand of the former from ethylene sources may be reduced five years hence by using acetylene as the raw material.

Ethyl alcohol is cited as the other main chemical from ethylene. This, too, is a source of raw materials for other processes. Production figures are again given, and total estimated requirements of ethyl alcohol from all sources for 1962 is stated to be 431 million gal, against a present-day demand of about 300 million gal. It is pointed out that future trends in ethyl alcohol production from ethylene will be influenced by such factors as economics, politics, and past performances. In particular, the price of carbohydrate materials, as raw material, will be very influential. E. J. C.

341. Petroleum chemicals in South Wales. Anon. *Petroleum*, Dec. 1951, **14** (12), 330–1.—A description of the Monsanto factory at Newport, Monmouthshire, which manufactures styrene plastic, detergents, oil additives, preservatives, fungicides, and chlorinated diphenyls. Considerable use is made of oil products as raw materials, and other major raw materials are chlorine, caustic soda, phenol, and sulphuric acid. R. E. P.

342. Competitive and co-operative research in the American petroleum industry—Third Cadman Memorial Lecture. R. E. Wilson. *J. Inst. Petrol.*, 1951, **37**, 407–25.—The history of the development of catalytic cracking is used to demonstrate the incentives which have been effective in American petroleum research activities since the first world war and the manner in which competitive and co-operative research has been stimulated. The conditions needed for productive research are summarized, and the nature of co-operative research activities in petroleum being undertaken in the U.S. at present are outlined. A. R. W. B.

343. A new science of trouble shooting. L. A. Seder. *Industr. Engng Chem.*, 1951, **43** (9), 2053–9.—Ways are outlined in which statistical methods can be used for diagnosing process ailments by carefully planned experiments. E. J. C.

344. Locating sources of variability in a process. W. J. Youden. *Industr. Engng Chem.*, 1951, **43** (9), 2059–62.—A statistical technique for locating sources of variability is outlined and illustrated, using some data obtained in quantitative chemical analysis. E. J. C.

345. Application of tests for randomness. C. A. Bennett. *Industr. Engng Chem.*, 1951, **43** (9), 2063–7.—The study of runs and use of mean-square successive difference are useful methods in testing many types of non-randomness encountered in chemical data. Method illustrated with reference to average weekly coke yield from a works and to the analysis of a standard sample of uranium oxide. E. J. C.

346. Predesign cost estimates. A. J. Wells and S. A. Singer. *Industr. Engng Chem.*, 1951, **43** (10), 2309–11.—The importance of research groups for pre-design estimates is pointed out, and training received in such a group is cited as a very useful industrial background. A good case is advanced for short-cut methods in such initial stages of cost estimating. E. J. C.

347. Capital cost estimating. W. T. Nichols. *Industr. Engng Chem.*, 1951, **43** (10), 2295–8.—Discusses various methods of estimating plant costs depending on available information. E. J. C.

348. Preliminary estimating by selective unit costs. P. Jandrioevits. *Industr. Engng Chem.*, 1951, **43** (10), 2299–301.—It is pointed out that errors in preliminary cost estimates due to the omission of a major item may be more serious than errors in the items themselves. A typical summary of an estimate and overall costs for various types of equipment, buildings, plant utilities, etc., which can be used for preliminary estimating are given. All data refer to American standards. E. J. C.

349. Estimating construction costs in chemical process industries. T. W. Bean. *Industr. Engng Chem.*, 1951, **43** (10), 2302–4.—The necessary steps in preparing preliminary estimates of construction costs are outlined, and more detailed methods for a complete estimate are described by reference to advanced design information. It is pointed out that " carbon-copy " jobs are rare, and that ultimately accurate compilation of data in the light of experience is the best basis for future estimates. E. J. C.

350. Preliminary estimation of operating costs. R. D. Newton and R. S. Airies. *Industr. Engng Chem.*, 1951, **43** (10), 2304–6.—The importance of operating costs in determining earnings from capital invested in process plant is stressed. Operating costs are broken down and discussed separately. Data are presented graphically on the U.S. rise in costs of labour, equipment, and chemicals since 1939. E. J. C.

351. Inflation in production and operating costs. V. R. Bechtel. *Industr. Engng Chem.*, 1951, **43** (10), 2307–9.—The establishment of a market for a particular product is not necessarily sufficient to justify investment of capital in new plant. It is very important that a detailed estimate of full operating costs, maintenance, taxes, etc., should be compared against predicted sales returns to establish a profit margin. The latter must give enough to remunerate those who provide capital, as well as rewarding management. E. J. C.

352. Correlation methods applied to production process data. K. A. Brownlee. *Industr. Engng Chem.*, 1951, **43** (9), 2068–71.—The author states that although many process records are kept and may contain useful information, they are often not used to advantage. He takes as an example the variation of stack loss from a nitric acid absorption column which varies with the temp of the cooling water, and uses statistical methods to examine the data. E. J. C.

353. Statistical design in chemical experimentation. W. L. Gore. *Industr. Engng Chem.*, 1951, **43** (10), 2327–8.—Use of statistical methods in designing experiments to ascertain the effect of variables is illustrated by reference to reaction rates of polymerization. It is stated that the chemist has not yet fully appreciated the value of statistical design principles in planning experiments. E. J. C.

354. The " rationalization " movement in [Polish] petroleum industry. M. Borecki. *Nafta*, Dec. 1950, **6**, 337–40.—This movement, aiming to bring forth suggestions for improvements and better use of existing methods, started to work in [Polish] petroleum industry in 1948, and a significant role is allotted to it in the Six-year Plan. To provide stimulus for a flow of ideas it was necessary to break the barriers between graduates and workers. Inventors clubs were organized to exchange ideas and organize excursions to scientific institutions. For the future efforts are going to be directed into selected channels, original ideas popularized, and the movement will get official support. Further education will be provided through clubs and individually to " rationalizers," and competition between clubs should provide further stimulus. M. S.

355. Natural gasoline—today's equipment is tailored to gasoline plant's needs. Anon. *Petrol. Process.*, 1950, **5** (12), 1320–4.—A condensed review is given of reports presented at 25th Ann. Mtng, California Natural Gasoline Assn, Los Angeles, Nov. 9–10. Reports reviewed are : Cooling equipment, G. H. Dieter ; Pumping trends, P. Armstrong ; Perforated-plate tray towers, H. R. Marsh ; Compressors, refrigeration, W. G. Palmer ; Paints and their application, J. W. Tucker ; Maintenance of cooling equipment, H. Dresser. D. W. F.

356. Programme of petroleum convention. Anon. *Nafta*, Oct.–Nov. 1950, **6**, 334.—Gives a list of twenty papers to be read, eleven of which are published in this issue. M. S.

357. British engineering and welding equipment. Anon. *Petroleum*, Sept. 1951, **14** (9), 233–40.—A review of some of the more important new equipment of special

interest to the petroleum industry manufactured by British companies. It includes brief descriptions of various welding plants, pumps, valves, refinery equipment, diesel engines, etc. R. E. P.

358. We must educate new staffs for the petroleum industry. A. Waliduda. *Nafta*, Sept. 1950, **6**, 246–8.—Describes the desperate shortages of trained staff in the industry and steps taken to fill the gaps. These include technical secondary schools with a bias towards petroleum; of these few exist already. M. S.

359. Can Russia fuel a war? M. Sittig. *Petrol. Process.*, 1951, **6** (10), 1116–17.—Russian crude reserves, production, refining technology, synthetic and substitute fuel production are briefly reviewed. E. J. C.

360. Saturation seen in styrene demand. Anon. *Refiner*, 1951, **30** (10), 105.—A review of past and present demand and production of styrene monomer. It is expected to become more plentiful in the future. A. R. H.

361. Combustion. B. Lewis and G. Von Elbe. *Industr. Engng Chem.*, 1951, **43** (9), 1925–41.—A review covering the period late 1948-late 1950. Subjects reviewed include kinetics of gas-phase reactions, ignition, detonation, engines, gas turbines, burners, furnaces, etc. 834 references. E. J. C.

BOOK REVIEW

Combustion Flames and Explosions of Gases. Bernard Lewis and Guenther von Elbe. New York : Academic Press Inc., 1951. Pp. 795 + xix. $13.50.

The title of this book has been borrowed by Drs Lewis and von Elbe from their former monograph published in 1938, but the authors point out that it cannot be regarded as a second edition of the older book, since the text is entirely new with the exception of a few brief sections. During the last thirteen years the volume of published work on combustion research has been increasing rapidly, so that the older book by Lewis and von Elbe and the well-known book by Jost must now be regarded as inadequate.

As the authors remark " Fundamental research should ultimately contribute toward improved understanding and control of technical combustion processes, particularly in engines." In the past combustion theory has lagged behind engineering practice and empirical methods have been much used by engines and furnace designers. The development of the turbo-jet engine for aircraft propulsion and of rockets of various types as war weapons has led to increased resources being made available for research into combustion phenomena, and future developments may well be based on the results of such research. Lewis and von Elbe of the U.S. Bureau of Mines are themselves responsible for many outstanding contributions to the literature since the publication of their earlier book, and are particularly well qualified to survey and assess the mass of published work which has appeared in recent years.

Part I of the book deals with the chemical kinetics of the reactions between fuel gases and oxygen and considers in detail the hydrogen–oxygen, carbon monoxide oxygen, and hydrocarbon–oxygen reactions. In the case of the last-named, there is now some degree of agreement as to the main features of the reactions, although the details require further careful study in order to throw new light upon certain aspects of the mechanism.

Part II deals with flame propagation, and it is in the subject matter of this section that most progress has been made in recent years. The authors have drawn upon their own published work in writing this section, and in their treatment have taken care to correlate many different phenomena which have been studied. Such topics as stabilization of burner flames and quenching in channels of critical diameters have been related. In this section a chapter on flame photography and pressure recording is followed by a lengthy and comprehensive discussion of combustion waves in non-turbulent explosive gases. This deals with such subjects as flame stability, development of a combustion wave from a source of ignition, propagation of combustion waves in tubes, ignition studies, combustion waves in closed vessels, and methods of determining burning velocity. Lewis and von Elbe have adopted the term combustion wave to describe a zone of burning which propagates through a medium. The section also contains a discussion of combustion waves in turbulent explosive gases, including the relationship between the " turbulent " and the " laminar " burning velocities. The important practical problems of ignition and flame stabilization in high-velocity streams also receive attention. Of interest to gas engineers, furnace designers, and others concerned with burners will be the chapters on mixing and combustion in fuel jets, and on burners with primary air entrainment. The latter chapter deals particularly with the problems involved in the prediction of burner performance when the fuel gas is changed.

Part III deals with the thermodynamic aspects of combustion, and it is this section which shows least change from the earlier book of 1938. The calculation of thermodynamic functions from the term schemes of molecular spectra is discussed. An interesting chapter deals with temperature and radiation of the burned gas, in which particular attention is paid to the well-known spectral line-reversal method of measuring flame temperatures.

In Part IV selected technical combustion problems are discussed: industrial heating is considered briefly, followed by combustion in engines, together with a study of flame stabilization in the turbo-jet engine.

Appendixes provide useful data, for thermo-chemical calculations, upon limits of inflammability, and upon flame temperatures.

In a rapidly developing subject it is inevitable that some recent work should not be included, but the book undoubtedly provides the most comprehensive review of the subject available and will become a standard work. It is beautifully produced and bound, with 355 illustrations in the text.

The book appears to be essential for all libraries concerned in any way with combustion problems. R. L.

ADDITIONS TO THE LIBRARY

A Rapid Method of Analysis for Bituminous Road Materials. A. R. Lee, C. M. Gough, and E. H. Green. London : HMSO, 1951. Pp. 12. 1*s.* net (postage 1½*d.*).

Road Note No. 10 from the Road Research Laboratory describes a rapid method for determining the binder content of a bituminous road mixture. Results are comparable with the BS method, and can be obtained in less than 2 hours. The "carpet" type of mixture can be analysed in 30 minutes.

Factual Data 50. El Dorado. Arkansas : State of Arkansas Oil and Gas Commission, 1951. Pp. 63.

This comprises the report of the chief engineer to the Commission on the working during 1950 of the oil and gas reservoirs under State control. There were ten controlled fields employing pressure maintenance or water and gas injection. Detailed data concerning fifty-six pools is given.

British Standards. London : British Standards Institution, 1951.

B.S. 761 : 1951. Vertical Multitubular Boilers of Riveted Construction. Pp. 50. 6*s.* net, post free.

B.S. 1739 : 1951. Filter Flasks. Pp. 9. 2*s.* net, post free.

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B.S. 1802 : 1951. Steel Spring Washers. Pp. 11. 2*s.* net, post free.

B.S. 1798 : 1951. Symbols, Terms and Definitions for Gas Quantities in Reciprocating Internal Combustion Engines. Pp. 7. 1*s.* 6*d.* net, post free.

B.S. 1804 : 1951. Parallel Dowel Pins (Steel). Pp. 10. 2*s.* net., post free.

B.S. 1780 : 1951. Pressure Gauges. Pp. 55. 6*s.* net, post free. .

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THE INSTITUTE OF PETROLEUM

NOTICES, JANUARY 1952

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Associate Members and Associate Fellows .	1 1 0	3 3 0
Student Members	Nil	1 1 0

APPLICATIONS FOR MEMBERSHIP OR TRANSFER

The following have applied for admission to or transfer of membership in the Institute. In accordance with the By-laws, the proposals will not be considered until the lapse of at least one month after the publication of this *Journal*, during which time any Fellow, Member, Associate Fellow, or Associate Member may communicate by letter to the Secretary, for the confidential information of the Council, any particulars he may possess respecting the qualifications or suitability of the candidate.

The object of this information is to assist the Council in grading the candidate according to the class of membership.

The name of candidates' proposers and seconders are given in parentheses.

Application for Membership

ABOLFATHI, Yadollah, student, Birmingham University. (*F. H. Garner; F. Morton.*)

BAKER, Reginald James, civil administrator and secretary, Manabi Exploration Co. Inc. (*P. H. de Pass; C. M. Edghill.*)

BLANNER, Allan, engineer, Clyde Fuel Systems. (Denmark.)

BUSHE, Peter Dalwyn Scott, driller, Trinidad Petroleum Development Co. Ltd. (*H. A. Bennett; C. C. Wilson.*)

COTTER, William Hingston, reservoir engineer, Bahrein Petroleum Co. Ltd. (*R. Godfrey; G. Sell.*)

DE MESTRE, Richard Etienne, senior petroleum engineer, Bahrein Petroleum Co. Ltd. (*R. Godfrey; G. Sell.*)

EVERETT, Eric George, exploitation engineer, United British Oilfields of Trinidad Ltd. (*J. L. Andrews; C. A. Dimitriu.*)

FOSKETT, Charles William, Major, R.A.S.C. (*P. F. Ellis; E. A. L. Francis.*)

FROST, Eric, senior commercial assistant, Shell-Mex & B.P. Ltd. (*R. J. Bressey; T. C. Bailey.*)

FROST, John Kenneth, chief engineer, Regent Oil Co. Ltd. (*A. D. Maclean; E. C. Scott.*)

HAINES, Peter Anthony Trevor, petroleum engineer trainee, Bahrein Petroleum Co. Ltd. (*R. Godfrey; G. Sell.*)

HALL, Cyril Stansfield, research engineer, " Shell " Refining & Marketing Co. Ltd. (*C. D. Brewer; P. L. Orman.*)

HARRISS, Dismond Sidney Marshall, assistant chemist, Anglo-Iranian Oil Co. Ltd. (*J. W. Hyde; P. S. Wilson.*)

HETHERINGTON, John Alfred, process engineer, Bahrein Petroleum Co. Ltd. (*L. H. T. Walker; N. E. Watson.*)

HOLTER, James Edward, depot manager, United Lubricants Ltd. (*F. Dakin; F. Massey.*)

KILPATRICK, John, student, Heriot-Watt College, Edinburgh. (*H. B. Nisbet; W. B. Peutherer.*)

LAURIE, Robert Arthur, assistant to engineer, Eagle Oil Co. Ltd. (*G. C. Carr; W. R. Davies.*)

LEAVETT, Brian Charles, engineer, Esso Petroleum Co. Ltd. (*I. McCallum; J. B. Robinson.*)

LOCK, John Kirby, assistant engineer, Anglo-Iranian Oil Co. Ltd. (*R. Stansfield; J. G. Withers.*)

SMITH, Roderic Kennedy, petroleum engineer trainee, Bahrein Petroleum Co. Ltd. (*R. Godfrey; G. Sell.*)

WRIGHT, John Thomas Matthew, junior laboratory assistant, Esso Petroleum Co. Ltd. (*L. H. Cowell; J. N. Kemp.*)

POLLOCK, Matthew, technical adviser, Admiralty. (*E. J. Sturgess; W. K. Davies.*)

Transfers

CHIR, Vincent Joseph, refinery superintendent, Kuwait Oil Co. Ltd. (*R. Wilson; R. M. S. Owen*) (*Associate Fellow to Member.*)

COWAN, Harold, assistant chemist, Messrs. W. B. Dick & Co. Ltd. (*J. Romney; W. R. Steet*) (*Student to Associate Fellow.*)

CRAWFORD, Edmund Robert, chemist, " Shell " Refining & Marketing Co. Ltd. (*H. E. F. Pracy; E. Fay*) (*Student to Associate Fellow.*)

EVANS, Howard Saunders, assistant development chemist, National Oil Refineries Ltd. (*E. J. Horley; P. F. Ellis*) (*Student to Associate Fellow.*)

HIBBERT, Ernest Davy, assistant construction engineer, Messrs. Foster Wheeler Ltd. (*K. E. Humphrey; W. N. Hoyte*) (*Student to Associate.*)

MALPAS, William Ewart, chemical engineer, Trinidad Leaseholds Ltd. (*F. H. Garner; F. Morton*) (*Student to Associate Fellow.*)

NEPPE, Solly Louis, general manager and chemical engineer, Gummed Tapes (Pty.) Ltd. (*W. T Jarrett; A. Faickney*) (*Member to Fellow.*)

OWENS, Ronald Ellis, laboratory assistant, Shell-Mex & B.P. Ltd. (*V. F. Smith; B. Turner*) (*Student to Associate Fellow.*)

SANDERSON, Francis Harold, manager (Petroleum Stocks & Records), Messrs. Berry Wiggins & Co. Ltd. (*A. Evans; F. W. Martin*) (*Associate Member to Member.*)

SMITH, Jack Kitson, geophysicist, Seismograph Service Ltd. (*F. H. Garner; G. Sell*) (*Student to Associate Fellow.*)

WILSON, Alfred, engineer, Shell Petroleum Co. Ltd. (*W. S. Ault; H. Hyams*) (*Associate Member to Fellow.*)

WILSON, Brian Sydney, research chemist, Lobitos Oilfields Ltd. (*V. Biske; E. Bowes*) (*Student to Associate Fellow.*)

NEW MEMBERS

The following elections have been made by the Council in accordance with the By-laws.

Elections are subject to confirmation in accordance with the By-laws.

As Fellows

GARDINER, G. L. ROUSSEAU, P. E.

As Members

CARTMEL, B. S. SYRENGHELAS, G. C.

As Associate Fellows

Bolt, J. A.	Jewell, J. W.
Chastell, L. E.	Jones, P.
Clark, C. E.	Laval, R. L. D.
Davies, R. H.	Slade, H. W.
French, K. H. V.	St. Konstas, A.
Griffiths, J. G.	Watson, A.

Transferred from Associate Member to Associate Fellow

Shiels L. O.

Transferred from Students to Associate Fellows

Fothergill, C. A.	Jones, G.
Hopkins, E.	Spottiswoode, J. N.

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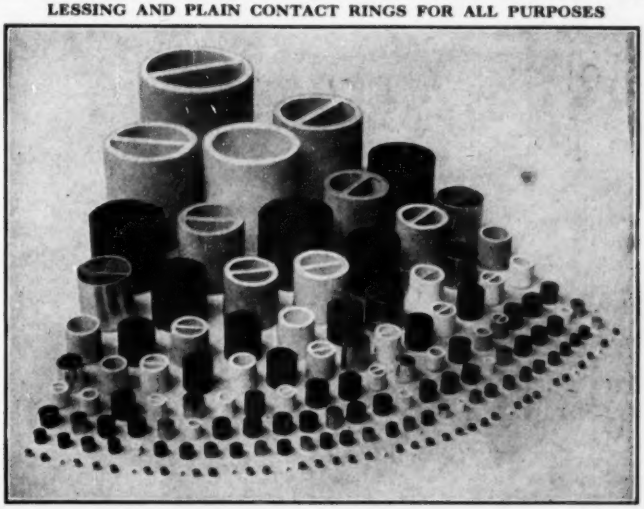

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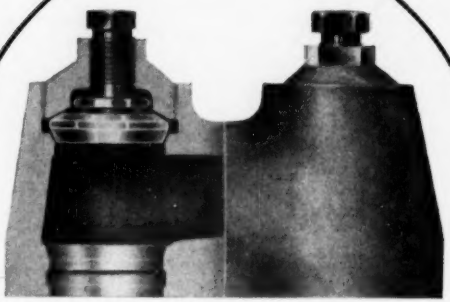

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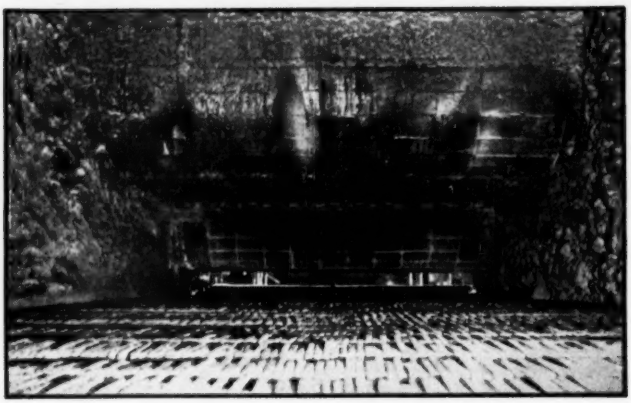

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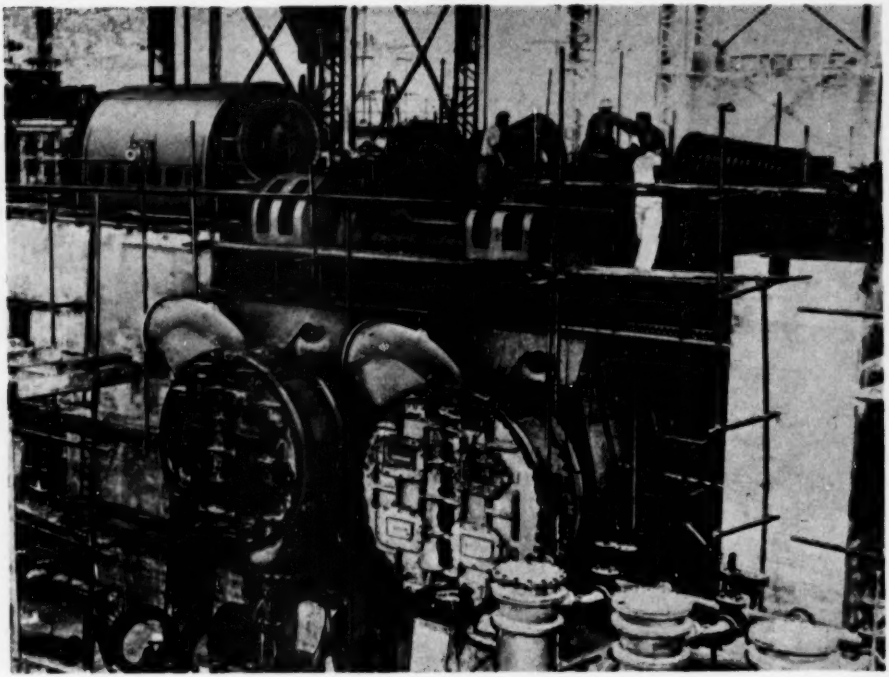

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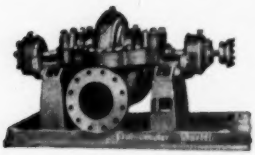

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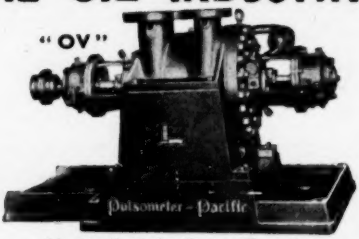

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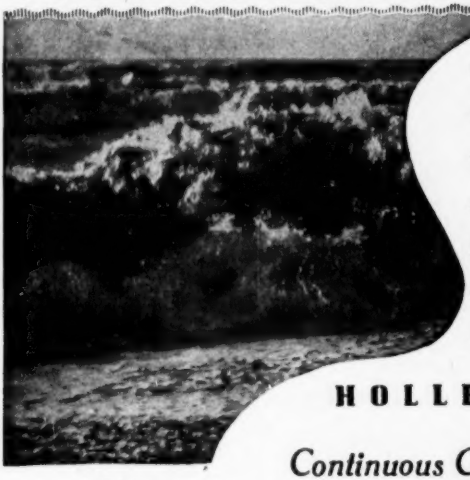

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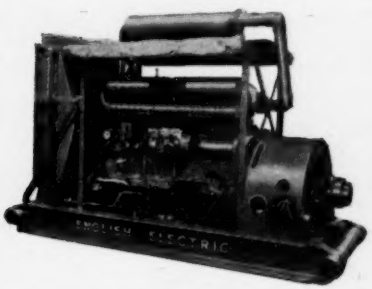

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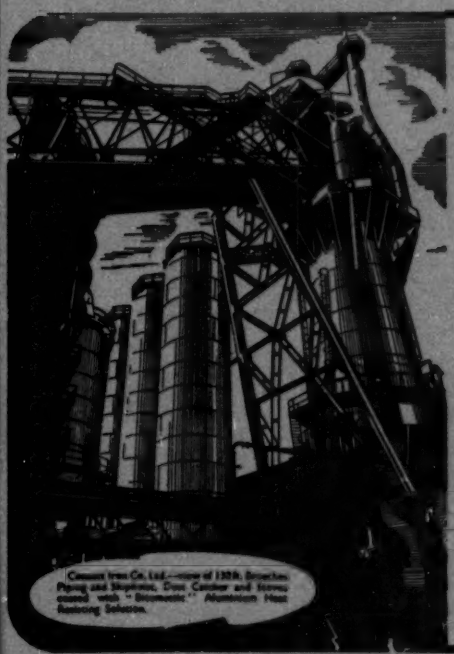

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